OXIDATIVE RANCIDITY OF PEANUT BUTTER SLICES UNDER DIFFERENT PACKAGING AND ATMOSPHERIC CONDITIONS

By

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Bachelor of Technology

Indian Institute of Technology

Kharagpur, India

1997

Submitted to the Faculty of the
Graduate College of
Oklahoma State University
in partial fulfillment of
the requirements for
the Degree of
MASTER OF SCIENCE
May, 2001

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Thesis Approved :

Thesis Advisor

Dean of Graduate College

ACKNOWLEDGEMENTS

I am heartily grateful to Dr. Danielle Bellmer for her patient guidance, timely advise and friendly support all throughout my study as well as stay in the department. I am also sincerely thankful to Dr. Gerald Brusewitz who's always been someone who I could look up to in matters concerning academics or otherwise for valuable advice. Not to mention, if it had not been for him, I could have never had the opportunity to do my Masters here at Oklahoma State University. I would always cherish the support of my two advisors in all that I did during my stay here in this school.

I wish to thank Dr. Tim Bowser, the third member of my committee, for so willingly attending to my presentations, and helping me with his suggestions as and when required. I also wish to thank him for his patient understanding during finalizing my thesis.

I'd like to express my sincere gratitude to Dr.Carla Goad, Department of Statistics, for her enthusiastic help in the analysis of my data, and for taking time off her busy schedule for my thesis.

I would like to thank Dr. McGlynn for his approval in making the Fruits and Vegetables Laboratory available for all my experiments. Dr. Gilliland also deserves hearty gratitude for allowing me to use his humidity chamber.

I wouldn't forget the help that I received from Lynn and Vijji in the initial days of my experiments – thanks to them for telling me the right ways to do my work and bearing with me for all the mistakes I made.

Besides, I would take this opportunity to thank all my colleagues and friends in the Department of Biosystems Engineering – Yu Zou, Missey, Bernadeth, Shekhar, Sridhar, Alicia, Bruno and Roshani, who made my stay in the department all the more special by being there for me whenever I needed them, to share a smile with.

A special note of thanks to Barbara for helping me out with every little problem that I've had, for that smile on her face no matter what time of the day I went to her with my complaints. I would also like to thank Gloria, Marge and Hope for their support every time I needed them.

Finally, to say the least, I'm eternally thankful to my parents for their being there for me, for the love and unconditional encouragement that they've given to me and for their prayers for my success in every step I took all the way. Also, I would like to make use of this time to thank Santhosh, my fiancé, for making each of my days with his thoughtful and caring ways and my dear friend Sharbari, whose unselfish friendship reflects in my life in so many ways.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Figures **Page**

CHAPTER (1)

INTRODUCTION

Peanut butter is a mainstay in the American diet. About 40 million North Americans use peanut butter everyday, with a total consumption of 800 million pounds per year (United States Consumer and Marketing Service, 1972). It is primarily used for its nutritive value, which is due to the high protein content of peanuts. Peanut butter is used for both individual retail as well as institutional use, such as meals in public schools, meals in prisons etc.

In the interest of convenience, a new peanut butter slice product has been developed. Peanut butter, as one finds it in stores now, lacks the convenience of other ready-to-eat products. An individually wrapped, single slice of peanut butter could be very beneficial in terms of easy use on sandwiches or crackers. The major selling points of a slice of peanut butter are convenience of use, easy transportability, portion control and better hygiene due to less contamination, as each slice is used only once. Also, storage of peanut butter slices is easy as it consumes less space and can be stacked one above the other.

The peanut butter slice formulation which has been developed consists mostly of peanut butter with a minimal addition of ingredients. It has a flavor and color nearly identical to peanut butter, and is easily peelable from the wrapper. The product has been successfully produced in a pilot plant setting on the same slice-forming equipment which is used to produce individually wrapped cheese.

Packaging of peanut butter has evolved from glass jars to plastic containers over the last two decades. The features of the various kinds of packaging have also been improved upon to ensure longer shelf-life and provide a higher quality product.

The present study is focused on the shelf-life characteristics of peanut butter slices under different packaging and storage conditions. Since a peanut butter slice has a greater surface area of exposure to the atmosphere, it is more prone to degradation by components like oxygen and moisture than a bulk sample of peanut butter in a closed container. Since peanut butter is a high fat product, it is highly prone to oxidative rancidity. Therefore, it is necessary to study its shelf-life characteristics under various conditions of storage and determine the suitability of different packaging materials to ensure greater shelf-life of peanut butter slices.

Objectives

The main objective of this study was to investigate the oxidative shelf stability of peanut butter slices when subjected to different kinds of packaging and storage conditions. The specific objectives were :

1. To evaluate the performance of various packaging materials with respect to rancidity of peanut butter slices.
2. To study the effect of temperature and humidity on the storability of peanut butter slices.

CHAPTER (2)

LITERATURE REVIEW

Peanut Butter

Peanut butter is a mixture of ground roasted peanuts and other ingredients such as hydrogenated oils, salt and sweeteners. After grinding and mixing, these ingredients are cooled and packaged at room temperatures. The packaging should be able to provide substantial resistance against oxidation of fats, since peanut butter is very high in fat content. Sometimes, it is packaged under inert gas to reduce the content of oxygen in the headspace to increase the shelf-life. Moisture in the product can be really degrading to the shelf life of peanut butter, since it turns the color of the product a dark shade of brown and affects its flavor negatively (United States Consumer and Marketing Service,1972).

Originally, peanut butter was ground and stored in a paper pail for retail consumption. Its sales increased after processors started to pack it in glass jars. The earlier glass bottles used to pack peanut butter had a metal screw cap that had a vinyl-coated pulp-board liner (United States Consumer and Marketing Service,1972). Some manufacturers used a glassine membrane over the mouth of the jar to provide greater resistance to oxidation. In recent years, the glassine membrane has been replaced by a foil/LDPE (low density polyethylene) laminate seal as a measure of tamper evidence. During the early 1980's, the metal cap was replaced by plastic caps, primarily injection molded polypropylene (PP).

In 1987, many of the leading peanut butter manufacturers switched to plastic jars made of PVC (Poly-Vinyl Chloride) or PET (Poly-Ethylene Terephthalate).

The PET jars were made by stretch blow-molding of injection-molded molten polymers, whereas the PVC jars were made by extrusion blow-molding. Although molding costs for PVC are lower, the greater strength and clarity of PET has resulted in a higher market for it (United States Consumer and Marketing Service,1972).

Plastic jars have been used to pack peanut butter for nearly 15 years. However, the plastic jars posed closure problems sometimes, as the plasticizer in the PVC gasket migrated into the plastic jar rim, bonding the lid to the jar. This caused unwanted locking of the jar cap. The current solution to this has been obtained by using an injection-molded polypropylene cap and a laminated plastic/foil membrane that was wax-bonded to a pulp-board liner in the jar cap. After application to the jar, the cap is sent through an electromagnetic field to produce an eddy current that induces heat in the aluminum foil in the plastic/foil membrane, and thereby heat seals the foil membrane to the jar rim. At the same time, it also melts the bonding wax to release the membrane from the pulp-board backing. It is crucial that the foil membrane be in intimate contact with the mouth of the jar and that the lid be screwed with enough torque to assure that the seal is leak-proof (United States Consumer and Marketing Service,1972).

This system provides an excellent barrier to both oxygen and moisture, and also provides a good tamper-proof seal. The polypropylene (PP) cap protects the foil seal until it is peeled off by the consumer. Then, it becomes an excellent method of protecting the food from oxidation until all of the product is utilized.

Development of Peanut Butter Slices

The current peanut butter slice formulation was developed by Dr. Danielle Bellmer at Oklahoma State University through much trial and error. The actual

ingredients used will not be disclosed here in order to protect the intellectual property rights. The viability of manufacturing individually wrapped peanut butter slices was tested using cheese slice manufacturing and packaging equipment at American Dairy Brands plant in Plymouth, WI. The molten peanut butter slurry is pumped into a slice forming machine where the individual slices are formed and heat-sealed (Fig.1). The strand of slices is then immersed in a cold bath to allow them to set (Fig.2). The cooled slices are then cut and stacked in rows from where they enter the packaging machine (Fig.3). The cheese slice forming equipment (KUSTNER Inc.) worked well for production of the peanut butter slices.

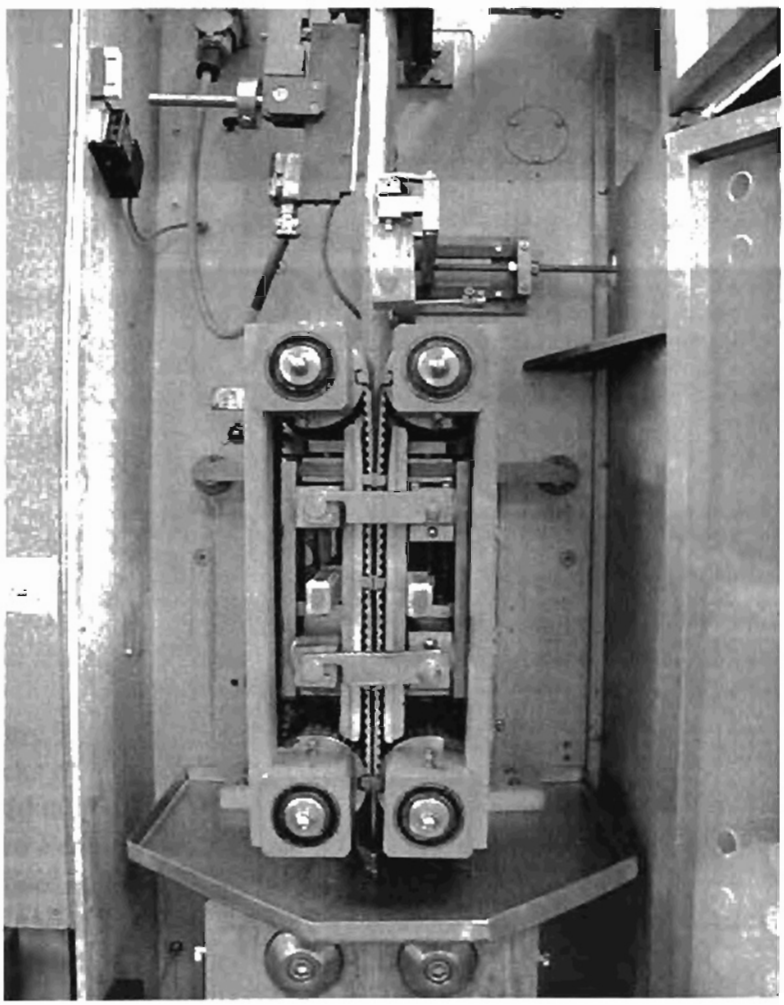


Figure 1.
Peanut Butter Slice
Batter Entering the Slice
Forming Equipment

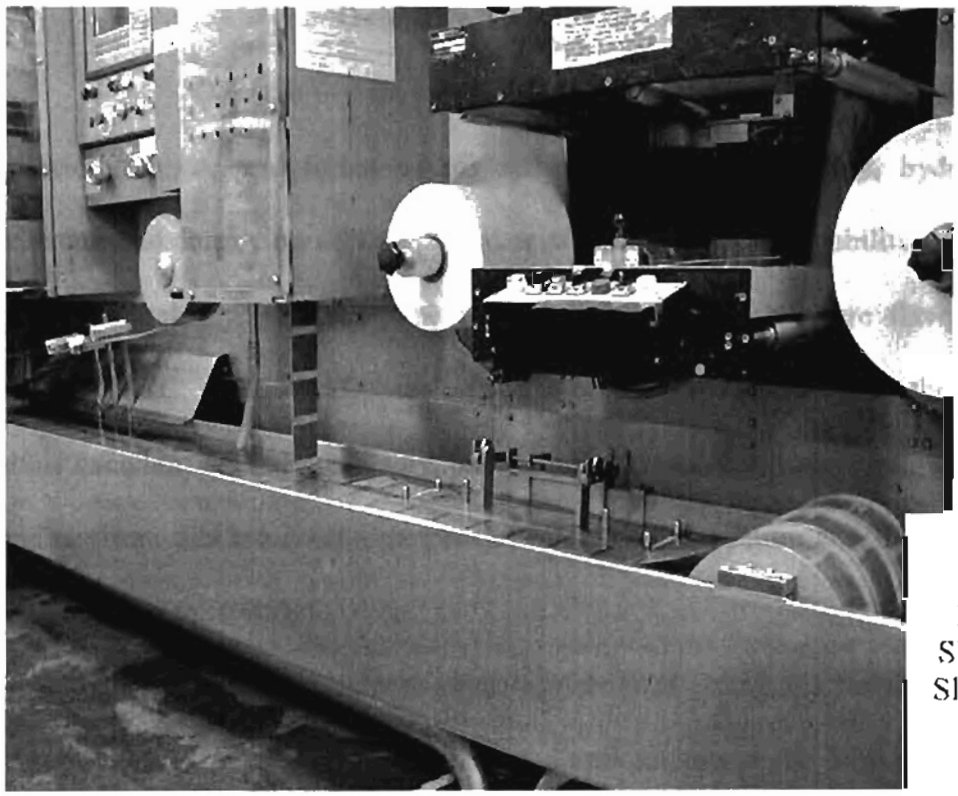


Figure 2.
A Strand of Peanut Butter Slices Exists the Slice Former and Enters the Cooling Bath

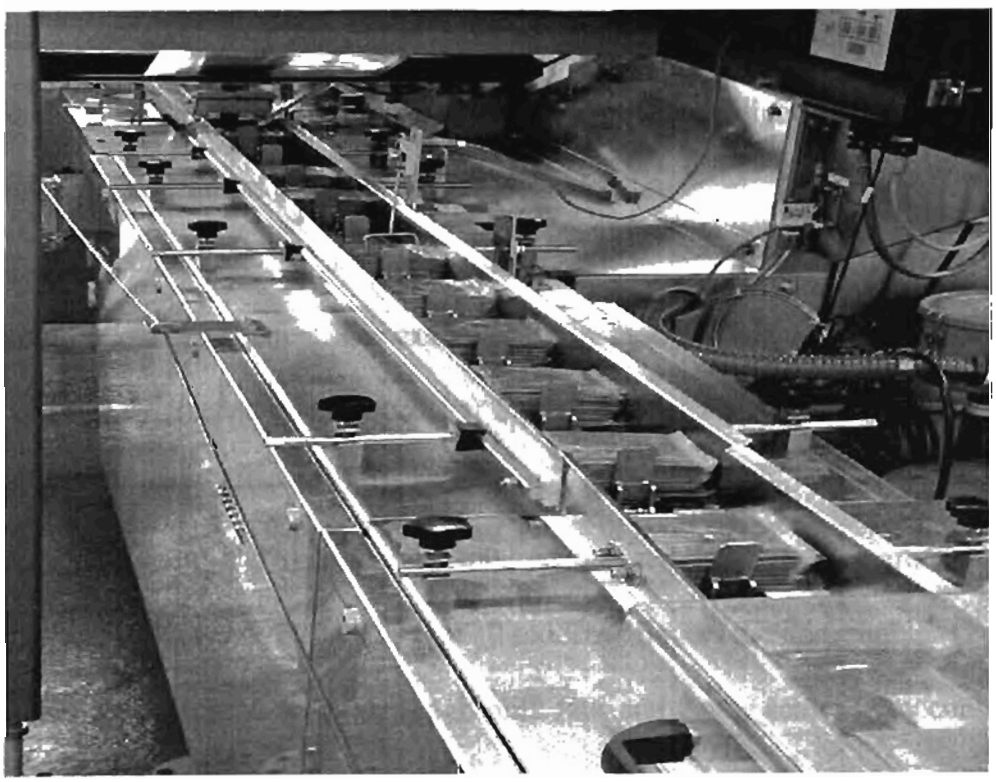


Figure 3.
Stacks of Individually Wrapped Peanut Butter Slices in the Packaging Equipment.

Oxidative Stability of Fats and Oils

Oil stability is the resistance of oils to chemical change or physical deterioration. A few forms of oil stability are color stability, hydrolytic stability, resistance to formation of foams, flavor stability, emulsion stability, heat stability and oxidative stability. Of these, the two most important kinds are flavor and oxidative stability. Most of the time, these need not be proportional to each other, i.e. an oil can show excellent oxidative stability, but mediocre flavor stability, or vice versa. Cottonseed and soybean oils are good examples of oils whose oxidation and flavor stabilities don't match (Smouse, 1985).

The main factors or components affecting oil stability are presence of phospholipids, soaps, enzymes, metals and antioxidants in the food product. The storage conditions of the oilseeds prior to processing and after processing, deodorization time, temperature and cool down rates contribute to the oil stability too. Fatty acid composition of the oil in the product, pigments in the food and exposure to light also determines stability of fats and oils (Smouse, 1985).

The content of fat, and therefore its oxidation, affects the texture and flavor of a product to a great extent. For example, if any kind of cheese is stripped of its fat content completely, the product that is left behind is tough and rubbery (Min and Smouse, 1989). The fatty phase in milk accumulates the flavor components that form during ripening of cheese, and therefore the oils centrifuged from cheeses are rich in cheese-like flavors. However, it is not true that all oxidized flavors are undesirable. There are toffee flavors which depend on oxidation of milk fats, and that is achieved by contact

of the fats with copper kettles. Also, the very popular Swiss cheese derives its typical flavor from oxidation products (Min and Smouse, 1989)

Fats, like many other unsaturated substances, undergo spontaneous oxidation. This leads to rancidity, and in turn, to loss of palatability and unwanted colors and odors. Products containing a higher proportion of unsaturated fatty acids are more prone to oxidation than those with lesser amounts. This is due to the fact that oxygen reacts more readily with unstable double bonds than with stable single bonds (Enig et al., 1983). However, this instability can be controlled using proper processing techniques, such as the use of antioxidants.

Mechanism of Oxidation

Considerable evidence exists to prove that the first step in oxidation of fats is the addition of oxygen to the *alpha* carbon atom at the double bond in a fatty acid chain. This causes the formation of hydro-peroxides. Although peroxides are the primary products of lipid oxidation, they break down and form a range of secondary products like peracids, aldehydes, ketones, ethylene oxides etc. (Hamilton and Rossell, 1986). During the early stages of oxidation, there is little apparent change in the composition, flavor or odor of the product, except that the concentration of peroxides changes within it. With extended storage time, the peroxides further breakdown to produce other volatile compounds that give rise to unwanted odors and off-flavors (Schaich, 1995).

During the process of establishing methods to test the stability of fats with respect to oxidative rancidity, various changes in the content of free fatty acids, iodine value, saponification value and content of oxidized fats were correlated to provide indices

of fat oxidation (Sanhueza et al., 2000). In the overall evaluation of fat stability, the following two criteria were discovered to be essential :

1. A test to indicate the condition of fat at the time of examination.
2. A test to indicate the length of time that the fat may be expected to resist oxidation.

Tests for Rancidity Analysis

Several different tests exist for the evaluation of oxidative rancidity in foods. Some of them are discussed below.

The Kreis Test :

This is one of the most primitive tests used to detect rancidity. In this test, a reagent called phloroglucinol reacts with a fat constituent, and the intensity of color produced by their reaction is said to indicate the degree of rancidity of the fat in the product. The procedure involves mixing a definite weight of the product with 0.5% phloroglucinol in amyl acetate, and adding trichloroacetic acid in amyl acetate to the test tube. The tube is then placed in a water bath at $45^0 \pm 0.1^0$C for 15min with vigorous stirring. After the tube is removed from the water bath, more cold trichloroacetic acid is added along with amyl acetate to stop the reaction, and bring the color of the sample within range of the instrument that is used to measure the color intensity. Any instrument may be used to measure the color (Mehlenbacher, 1960). A blank sample is tested simultaneously in the same way, and the corrected value of the sample is obtained by subtracting the blank reading from the sample reading.

There is a considerable amount of controversy over the efficiency of this test. As concluded by Powick (1923), the compound epihydrin aldehyde accounts for a

positive Kreis test, whereas Deatherage and Mattil (1939) believed that homologues of this aldehyde might also cause the Kreis test to be positive. It was found by Holm and Greenbank (1924), that the color intensity is proportional to the amount of oxygen absorbed by the fat, which need not be a direct measure of its rancidity. The development of color is not a direct measure of the development of rancidity, and sometimes fresh samples give rise to some colors when they react with Kreis reagent.

Permanganate Reduction Method:

This test measures the stability of fats in a food product. It was devised by Grettie and Newton (1931), and involves the passage of air first over a heated sample of food containing the fat component and then into standardized potassium permanganate solution. The fat is dispersed on strips of filter paper in glass tubes maintained at high temperatures in a water bath during the induction period of the fat. In this process, the potassium permanganate is reduced by the volatile oxidation products from the fat, and the degree of its reduction provides an index of fat stability.

This test is mainly used for the analysis of dry products containing fats like cereals. This was the test used by Horne et al. (1948) to test the stability of commercially produced army ration biscuits.

The procedure involves grinding the sample so that it has a uniform particle size. A fixed weight of the ground sample is taken in an open tube and both of the ends are closed with cotton to keep the sample in place. The sample is inserted into a water bath at 100^{0}C and washed and dried air is passed over it. Then, this tube is taken off the water bath and replaced with a tube containing standardized potassium permanganate solution. Finally, both, the sample and the potassium permanganate

solution are transferred into an Erlenmeyer flask and 5% potassium iodide is added to the mixture. The unreacted potassium permanganate solution is titrated with 0.005N sodium thiosulphate solution. The difference between the volume of potassium permanganate in the tube and volume of sodium thiosulphate used for titration gives a direct measure of the potassium permanganate reduced by the oxidation products of the fat sample (Mehlenbacher, 1960).

When the volume of reduced permanganate solution is plotted against the aeration time, the length of time required to reach the point where significant change occurs in the rate of oxidation is the Induction Period. Induction Period is the time required for oxidation to start after all the antioxidants or inhibitive sources are used up in the food product.

Schaal or Oven Test Method

In this method of analysis, an adequate amount of the sample is placed in a beaker and held in an oven at a warm temperature (70^0C) until rancidity sets in. A regular time interval is maintained between testing the samples depending on the quality of the fat in the product. The end-point is determined by an organoleptic evaluation of the odor of the product. The peroxide values of the samples in storage may be measured, but the results from Peroxide Test, discussed later in the dissertation, are more accurate by the Active Oxygen Method than by this method.

This method is most commonly used for bakery products. One major limitation of this method is its subjective nature (Mehlenbacher, 1960).

Photochemical Methods

Color indicators are sometimes used to determine oxidation/reduction of fats. Indicators such as methylene blue and eosin-erythrosine are commonly mixed with a fatty substance and the rate of color reduction, which indicates the rate of oxidation of fat, is measured with a photoelectric cell. From the studies of Royce (1933), it was found that in this method, the end-point was unsatisfactory due to the secondary color reactions that occurred in the mixture.

This method is used to study the stability of vegetable oils. As a general observation, the natural fluorescence of vegetable oils decreases with extended storage time as oxidation proceeds (Mehlenbacher, 1960).

Active Oxygen Method

This method was introduced in the 1930's (Mehlenbacher, 1960). It is the most popular test used to determine fat stability. This method is based on the formation of compounds containing active oxygen when fats are oxidized, which can further be reduced by potassium iodide. This is done by heating the substance at elevated temperatures during which the sample is simultaneously aerated with washed air. Heating and aeration are continued until the peroxide content reaches a pre-determined value. This value is fixed to approximately match the point where rancidity starts in various kinds of fats.

Peroxide Test

The primary product formed by the oxidation of any lipid is a hydro peroxide. In the peroxide test, the food containing fat is subjected to titration with sodium thiosulphate to neutralize the peroxides formed. The quantity of sodium thiosulphate

required to neutralize all the peroxides formed determines the extent of rancidity developed. The end point of titration is established by a sharp color change in the starch indicator that is used.

Since peroxides are formed in any kind of fatty foods when rancidity sets in, the peroxide test is one of the most widely used rancidity tests. However, at later stages of oxidation, the peroxides finally break down to form other components (Woodroof,1983). The peroxide value (P.V.) is a measure of the amount of peroxide in rancid oil expressed in milli equivalents of oxygen per kilogram of food product. If P.V. is plotted against time, the curve exhibits a gradual rise and then a steady fall indicating the breakdown of peroxides into other secondary components. Hence, a low P.V. doesn't necessarily mean absence of rancidity (Lennersten, 1998).

Thio Barbituric Acid Test

The secondary products of oxidization of unsaturated fatty acids consist of aldehydes and other degradation products. In this test, the aldehydes react with a reagent called TBA/TCA (Thio Barbituric Acid/ Trichloro Acetic Acid) upon heating to produce a red color which is due to the formation of malondialdehyde (Bernheim et al., 1948). The intensity of the red color is measured using a spectrophotometer and that provides an index of the degree of rancidity.

The advantage of this method is that the reagent causes no separation of fats from the food product unlike many other reagents that are used to test rancidity (Guzman and Vicario, 1998). However, the red color formed in this test may also be partly due to the reaction of the reagent with other aldehydes which form by the breakdown of the non-fat components in foods, thus interfering with the results.

The tests that were used for rancidity evaluation of the peanut butter slices were the Peroxide Test and the Thio Barbituric Assay. These were chosen because they are the most widely used tests for high fat foods, and peanut butter is high in fat content. These tests are described in detail in Chapter(4).

Any fatty product may be subjected to shelf-life studies in order to determine the time at which rancidity first develops. Accelerated tests may be used to determine the effect of a particular parameter on the stability of the product, but in this case the actual rancidity is not measured (Anderson and Jones, 1999).

Antioxidants

Antioxidants inhibit the oxidation of fats. In the initial stages of oxidation, any antioxidants present in the oil play a role in inhibiting oxidation. This can occur by the removal of dissolved oxygen or by the neutralization of free radicals formed during auto-oxidation (Chu, 1991). Auto-oxidation in oils takes place primarily by branching chain reactions.

In either of these forms of inhibition, the antioxidants are eventually used up. It is then that the oxidation process is accelerated. The initial stage, when auto-oxidation doesn't occur, or proceeds slowly, is called the Induction Period, during which oxidation is rather slow. In the Peroxide Test, if the Peroxide Values are plotted against time during this period, the Induction Period can be easily determined by the sudden increase in slope of the graph at the end of this period.

Some studies have been conducted to evaluate new methods of determining the oxidative stability of fats in foods and the effectiveness of natural

antioxidants. In recent years, the use of natural antioxidants has gained momentum due to a global trend to avoid the use of synthetic food additives (Frankel,1993).

Many edible fats contain natural antioxidants, the most common of which is tocopherol. But a portion of the natural antioxidants are removed during processing of the foods. This loss can be made up using external antioxidants. For the antioxidants to perform their role, it is essential that the antioxidant be completely soluble in the fat and that it shouldn't add any flavor, smell or color to the food product even after prolonged storage. Also, it shouldn't have any detrimental physiological effects and there should be no significant chemical changes upon heating of the product. The antioxidant should retain its inhibitive properties even after use of the fat in or with any other food (Emanuel and Lyaskovskaya, 1967).

The mechanism of inhibition follows a chain reaction between trace amounts of the antioxidant and the substrate until the rate of the reaction increases toward termination. This could otherwise be called 'chain termination'. It has been found that a substance that might act as a very effective antioxidant on one substrate, may be completely inert on another (Emanuel and Lyaskovskaya, 1967).

Butylated Hydroxytoulene (BHT) and Butylated Hydroxyanisole (BHA) are the most commonly used antioxidants. Since any one substance is not fully capable of meeting all the conditions of a good antioxidant, it is a common practice to use antioxidant mixtures.

BHA serves as a very good antioxidant for stabilizing fats in pork, turkey, many fish oils, vegetable oils, orange juice and groundnut oil. BHA mixtures are also

used to stabilize Vitamin A, dough products, potato chips, wheat and corn flakes etc. (Emanuel and Lyaskovskaya, 1967).

BHT is not only used in the food industry, but also is used to stabilize waxes, resins, greases, oils, plastics, etc. BHT, either by itself, or when mixed with other antioxidants, reduces fat oxidation in milk, butter, lard, olive oil, cottonseed oil and shortening, beef and pork. It also protects carotene in animal fats from oxidation (Miyazawa et al., 1991).

Packaging of Foods

Packaging of foods in various forms has been a prevalent practice for a very long time. The practice began with water, and gradually extended to other products because it kept them dry and clean, improved transportability, kept them safe from insects and germs and inhibited the spoilage of the food products by oxygen, moisture and light. The modern packaging era began with the advent of canning in the early 1800s (Briston and Katan, 1974). Better packaging materials came into the picture as new preservation techniques evolved to expand the variety of more appealing, safe and nutritious foods that could be transported over long distances and stored over extended periods of time before consumption.

Different food products are packaged in different forms using different kinds of materials, depending on the properties of the material as well as ease of use. Metal-capped jars, tinned steel, enamelled tin cans, corrugated paperboard, coated paper and aluminum foil are chiefly used, along with a huge range of plastics in food packaging. The many versatile properties of plastics make them ideal for packaging foods. In most cases of usage of plastics, it is a combination of properties that is put to

use. Plastics exhibit several useful characteristics which make them good for food packaging.

Properties of Plastics

Densities of most plastics are very low. A range of density from 0.9 to 1.4 g/cc is typical of most plastics used in packaging. Due to the thin gauges commonly used in packaging, rigid plastic containers resist breaking much more effectively than glass. This, along with their low density, makes them more desirable for packaging over glass, which is otherwise an excellent material from the standpoint of food safety. Also, plastic containers do not pose the hazard of sharp edges that can occur with glass (Jenkins and Harrington, 1991).

Most plastics are flexible, and all flexible packages are easy to seal. During sealing, a coating on the flexible substrate is heated within the packaging equipment until it melts, and then is held in contact with the similarly coated opposite side until the two coatings solidify as one layer. Most plastics can be easily formed into thin, strong and clear films. Plastics are unsurpassed in the ease with which special shapes can be readily created (Jenkins and Harrington,1991).

Plastics don't rot or rust unlike many other materials. They are also impervious to attack by most degradation factors, except ultraviolet rays. Plastics also provide considerable barrier to moisture and oxygen. Most foods are sensitive to water vapor and oxygen. Glass and aluminum foil are totally impermeable to these, whereas plastics or polymer coated cellophane have much higher permeabilities. However, they rank above uncoated paper. Some of the plastics have sufficiently high moisture barrier

that is adequate for most foods. In order to achieve a high oxygen barrier, a multi-layer construction of plastics is normally adopted (Jenkins and Harrington,1991).

On the other hand, the finite permeability of plastics sometimes becomes a positive attribute for products like freshly harvested fruits and vegetables. The shelf-life of such products can be greatly enhanced if they are packaged in a material that is selectively permeable to oxygen and carbon dioxide (Czerniawski et al., 1997). Most plastics also provide an excellent barrier against contamination from odors from the environment.

Plastics have an advantage over other packaging materials in terms of lightness and appearance. Unmodified plastic films and sheets range in appearance from crystal-clear to hazy. Pigments or soluble dyes can be added to make them totally opaque in any color or to make transparent films and sheets. Plastics can be printed on but paper and cellophane are marginally superior to plastics in this regard. Most plastics require an inexpensive pre-treatment to develop this attribute, while it is expensive to print on paper or cellophane. Metal and glass are more difficult as well as expensive to print on. Use of packaging to advertise and add to the aesthetics of the product is extremely important in the food industry. Very thin coatings of aluminum can easily be vapor-deposited on plastic substrates in vacuum chambers. This is usually used to provide barrier to light, moisture and oxygen (Jenkins and Harrington,1991).

Most of the new plastics do not add flavors, nor do they extract essential flavor components from foods. Most plastic films are more resistant to tearing and puncture than aluminum foil. Moreover, they also resist cracking, which is very common in aluminum foil. Plastic films remain flexible at low temperatures, unlike cellophane

which becomes brittle below 40^0F. These traits of plastics make them popular materials for packaging over several other materials that were used in the past (Jenkins and Harrington, 1991).

Permeability

Permeability of the packaging material with respect to moisture, oxygen or any other component that might contribute to deterioration of the food product is a very important parameter in determining the suitability of a particular material as a good packing material. Permeability is expressed in terms of volume or weight of the degrading component that can diffuse through the packaging material per unit surface area of the material per unit time. These numbers give an estimate of the extent to which the packaging material can provide barrier against the degrading components.

Variables Affecting Permeability:

The chemical structure of a polymer influences its permeability as it determines the level of interaction between the packaging material and the permeate. The value of permeability changes for each different pair of polymer/permeate. An increase in polymer crystallinity (density), orientation or cross-linking, i.e: the morphology of the polymer, decreases permeability.

Humidity affects permeability (especially in hydrophilic polymers). Oxygen permeability increases with relative humidity for certain types of polymers, and decreases for some kinds like amorphous nylon. Permeability increases with increase in temperature. The equation that relates the permeability of a packaging material to the temperature is $P = P_0 e^{-E_p/RT}$, where P_0 is a pre-exponential term, E_p is the activation

energy, R is the gas constant and T is the temperature in Kelvin. The equation can be written in an alternative form, which is :

$P_2 = P_1 e^{Ep/R(1/T1 - 1/T2)}$, where P_1 is the permeability at temperature T_1, and P_2 is the permeability at temperature T_2 (Valentas et al., 1997).

Filler materials generally decrease permeability. However, the effect is complicated by the type, shape, amount of the filler and interaction with the permeant. There is no effect of concentration of the permeant on the permeability at low and moderate pressures for gases and low activity values for organic compounds in the range of Henry's Law (Perez and Krochta, 2001). Strong effects are found for organic compounds at high values of activity. Permeability is usually, but not always, increased by plasticizers.

In principle, film thickness does not affect permeability, diffusion coefficient or solubility. However, thickness of the film may generate different morphologies, say, by having different cooling characteristics during processing (Abe, 1994). Molecular weight of a polymer has been found to have little effect on the permeability of a polymer except in very low range of molecular weight (Marjanski et al, 1996). Table I shows a wide range of variability in the oxygen and moisture permeability of various plastics available.

Table I. Some Primary Plastics Used in Packaging and Their Permeability Properties

Plastic	Oxygen Permeability ($cc.\mu m/m^2.d.kPa$)	Moisture Permeability ($cc.\mu m/m^2.d.kPa$)
Cellophane	1.4 @ 0% RH 5.7 @ 76% RH	16000000
Polyethylene and Ethylene Copolymers	1900	59000
Polypropylene	620	44000
Polyvinyl Chloride (PVC)	29	180000
Polyvinylidene Chloride	3.3	6000
Ethylene Vinyl Alcohol (EVA)	0.15 @ 0% RH 0.3 @ 65% RH	110000
Polystyrene	1100	730000
Polycarbonates	910	910000
Polyacrylonitriles	3.5	420000

Source : Valentas et al., 1997

Examples of Food Packaging

Orange juice in aseptic cartons has gained tremendous market all over the world in the last few years. Aseptically packed juices exhibit high standards of quality, but there is considerable amount of flavor change during storage in aseptic orange juice. The polyethylene polymers in the aseptic cartons that come in direct contact with the juice have been found to accelerate oxidation, non-enzymatic browning and flavor absorption (Jeng et al., 1991).

Low density polyethylene bottles are used for products that need to be squeezed out, like ketchup.

Meats are shrink wrapped typically into triple-layered co-extruded films made of EVA copolymer, PVC/PVdC copolymer and EVA copolymer, sometimes with a polyamide as the barrier layer. The bags are evacuated before sealing using heated jaws (Robertson, 1993). The color and texture of meats are very prone to degradation by oxygen since myoglobin, the muscle pigments that render color to muscle fibers, are susceptible to oxidation rather easily. Hence, multi-layered materials are chosen to pack meats such that the combined oxygen permeability doesn't effect the quality of stored meats.

Milk is packaged in glass, coated paper or plastic containers for retail use. These materials are either used to make re-usable packs or as single-use packaging. Light destroys riboflavin in milk, and that is the reason why use of brown glass was proposed to make milk bottles. The color of the container determines the amount of light reaching milk. A study was conducted to determine the extent of deterioration in milk by light, and the materials used were clear polyethylene pouch, co-extruded laminated polyethylene pouch (with inner black pigmented layer), paperboard carton and plastic jug. Milk was found to develop off-flavors in all except the laminated pouch (Robertson, 1993)

Breakfast cereals need to be protected against degradation by loss of crispness, lipid oxidation, loss of vitamins, mechanical damage and loss of flavor. The grains that are used to make breakfast cereals are very high in unsaturated fats, and therefore the rate of lipid oxidation is high in these products in the absence of efficient packaging. These products are typically packed in fiberboard boxes lined with waxed glassine. A more recent trend is to use various plastic materials like thin gauge HDPE for lining the boxes instead of glassine (Robertson, 1993).

The two main effects of plastic packaging on fatty foods are stress cracking and migration (vom Bruck et al., 1981). It has been shown that there is a definite mathematical relationship between the concentration of an additive in the polymer, and the migration into the food material. Therefore, it is recommended that regulations for plastic food packaging are based on compositional limits, rather than migration limits due to specific advantages in compliance and control.

Packaging of Fatty Foods

Foods containing fats face the problem of oxidation of unsaturated fatty acids, which cause off-odors, off-flavors and untimely polymerization of the fatty acids. Thus, utmost care needs to be taken while packaging such foods to protect them against oxidation. Moreover, the packaging material needs to be oil-proof and free of components like lubricants or polymer modifiers that could be extracted or absorbed by the oil/fat (Michalski and Desobry, 1998).

There exists a kind of glass container developed by Foil Mate™ that has a seal impermeable to oxygen, carbon dioxide, water vapor and other undesirable gases. It has been developed by combining the impermeability of aluminum foil and the adherence of polymer films. These containers are suitable for dry products, as well as liquid and viscous products like fruit juices, sauces, honey, syrup, peanut butter, jams, jellies, mayonnaise, wine and tomato products (Poad.1982)

In the packaging of fatty products like peanut oil, the oxygen permeability of the packaging material plays a more significant role than the moisture permeability. Gopalakrishna and Prabhakar (1983) studied the effect of water activity on the rate of autoxidation on raw peanut oil at 37^{0}C. It was noticed that in three weeks of storage,

there was no significant formation of peroxides. The results indicate that there is a protective effect of certain components in peanut oil on the autoxidation at high rates of water-activity.

CHAPTER (3)

MATERIALS AND METHODS

Peanut Butter Slice Preparation

All the slices of peanut butter were made with the same formulation. The peanut butter used was 'Jiff Original' that was bought at the local WALMART superstore. Each slice weighed about 49gms and contained peanut butter, monoglycerides, polysaccharide gum, sugar and peanut flour. Each batch was prepared such that 12 peanut butter slices could be made each time. A water bath was set at a temperature of 100^0C. All the dry ingredients (peanut butter, monoglyceride, peanut flour and powdered sugar) were first weighed separately on plastic weighing dishes. To prepare the gum mixture, 115g of water was mixed with 5g of gum. The mixture was stirred and mixed thoroughly and then heated on a hot plate to a temperature of 80^0C. The mixture was stirred at that temperature for 5 mins. and then kept warm until use to prevent it from setting.

The monoglyceride was poured into a hot aluminum bowl on the water bath and allowed to melt completely. Peanut butter was next poured into the bowl and mixed well with the monoglyceride using the mixer for about 30 sec. Next, the gum mixture was weighed out of the beaker onto a weighing dish and poured into the bowl with peanut butter very quickly (i.e: allowing very little time for it to set or solidify). The peanut butter mixture was mixed uniformly for 4 mins. and powdered sugar and peanut flour were then added separately into the mixture. Mixing was continued for another 1 min. Care was taken to ensure that the total mixing time was at least 5 mins.

Finally, the bowl was lifted from the water bath and the bottom was wiped with a towel so that no water dripped into the slice molds. Definite portions of the mixture were poured into beakers that had been marked such that they could hold 40g of the mix for each slice. Each portion was then poured into the molds which had been arranged with a definite kind of packaging material beneath each of them prior to beginning the experiment. The molds consisted of teflon cutting boards in each of which six squares had been cut out, each of 76mm width.

The last two steps needed to be done with as much speed as possible to prevent the peanut butter mixture from setting before pouring it into the molds. After the peanut butter mixture solidified on the packaging material, the slices were cut out using a blunt knife, and the molds were removed. A sheet of the same packaging material was spread on the slices and each slice was cut out with a layer of packaging material on either side. The packaging material was sealed on all four sides of each slice using a heat impulse sealer. Each slice was color coded with respect to the packing material it was wrapped in, and the date on which it was made was labelled on the package. The Peroxide Test and Thio Barbituric Acid Assay were both conducted on the slices at '0' time period before the slices were packaged. These tests were done within 1-2 hrs after preparing the slices.

Chemical Analysis

Peroxide Value

Oxidation is the most common cause of rancidity. When an oil is oxidized, as discussed earlier, the first product formed is a hydro-peroxide. Therefore, the most common method of assessment of rancidity is by the determination of the peroxide value

(P.V.), reported in units of milli-equivalents of oxygen per kilogram of fat, which is a direct measure of the amount of fat that has been oxidized. This method is empirical and the results and accuracy of this test depend on the experimental conditions.

To conduct a test, 5 gm of peanut butter sample was taken in a beaker and manually ground thoroughly in a 30 ml mixture of 3:2 glacial acetic acid and chloroform solution. 0.5 ml of saturated potassium iodide solution was added to this mixture, as a result of which iodine was liberated due to reaction with the peroxide. This was then titrated against a standard solution of sodium thiosulphate, using starch solution as indicator. This procedure was followed but without any sample in the beaker to determine the titration value for a blank sample (Gordon et al., 1988)

The peroxide value is calculated as follows :

P.V. = (S – B) * N * 1000 / Sample Weight, where

S = Titration value of the sample (ml)

B = Titration value of the blank sample (ml)

N = Normality of the Sodium Thiosulphate solution 0.1N

Sample Weight = 5 gms

There are mainly two sources of error in this test. First, the absorption of iodine at unsaturated bonds in the fatty acid can lead to a low P.V. Second, the liberation of iodine from potassium iodide by oxygen in the sample can lead to a high peroxide value. Therefore care should be taken to ensure that the right amount of potassium iodide is added to the sample mixture (Hamilton et al., 1986).

Fats that have been stored for some time might show high peroxide values, though there might not be any undesirable flavor problems. This is because peroxides are flavorless. However, during prolonged storage, peroxides decompose into aldehydes and ketones, many of which have pronounced off-flavors (Ahn et al., 1998).

Thio Barbituric Acid Assay

The Thio Barbituric Acid (TBA) Assay is yet another empirical method to detect rancidity in fats and oils. This method relates to the level of aldehydes present in an oil. Aldehydes are formed during storage of fatty products as peroxides break down. The particular aldehyde that reacts in the TBA test is 'malondialdehyde'. It reacts with the TBA reagent to give a red chromogen, the intensity of which is measured using a spectrophotometer, which is an empirical measure of the amount of aldehyde in the rancid fat. But the limitation of this method lies in the fact that though there might be a considerable amount of aldehydes present in a fatty product after a certain period of storage, not all of them necessarily are a product of oxidation of fats.

For the experiment, 5 gms of the peanut butter sample was ground with 15 ml of double distilled water. One ml of the homogenate was taken and two ml of TCA (trichloro-acetic acid)/TBA reagent was added to it with 50 µl of BHA (Butylated Hydroxy Anisole). This solution was thoroughly heated for 15 min. in a boiling water bath, and then cooled for 10 min. in cold water. The mixture was then centrifuged at 2000G for 10 min. at 5000 rpm. The supernatent liquid was collected and its absorbance was read using a spectrophotometer at a wavelength of 531 nm against a blank sample. Finally, a TBA standard curve was constructed.

However, in this test, it is possible that other aldehydes in the product would react to form the red chromogen. Also, oxidized proteins and other food components might contribute to colored products on reaction with the TBA reagent. Therefore, the spectrophotometer reading need not be in direct proportion with the amount of fats oxidized during the occurrence of rancidity.

Experimental Design

The design of the experiment is based on evaluation of rancidity of the peanut butter slices under different combinations of temperature-humidity conditions and packaging materials. The atmospheric conditions chosen are based on the most probable conditions of storage and transportation. The packaging materials are selected based on common materials used in the market, specifically in the packaging industry to pack oxidation-sensitive food products. The major criteria in choosing the packaging material is its permeability to oxygen and moisture. Experiments were carried out over six months of storage time, with the first set of observations being taken after one week of storage and then tests were done every month.

The following storage conditions were chosen :

- Room Temperature and Humidity (25^0C ± 5^0C, 45%RH ± 10%)
- Refrigerated conditions (4^0C ± 1^0C , <20%RH)
- Oven Temperature (40^0C ± 1^0C, <20%RH)
- Humidity Chamber (40^0C ± 1^0C , 80% RH ± 2%)

These conditions were chosen keeping in view the range of conditions that the peanut butter slices might be subjected to during shipping and storage of the product during all weather and seasonal fluctuations. One point to be noted when drawing

conclusions about the results from the experiments is however that the temperature and humidity conditions were not strictly monitored , especially under room conditions, and therefore there could have been some fluctuations in storage temperature and humidity since the experiments were carried out from summer to winter.

The following packaging materials were used to test the slices :

- Saran wrap
- HB1, a high barrier material from Phillips (Bartlesville, OK)
- DK11, a low barrier material from Phillips used for packaging of fresh foods
- Cheese packaging material from Printpack, Inc. (Elgin, IL)

The basis for selection of these materials was to incorporate a wide range of oxygen and moisture permeability of materials. This would enable the determination of the extent to which permeability characteristics of the packaging material influences rancidity of peanut butter slices. Also, the selection of the materials was based on the most widely available and used polymers. For example, saran wrap is a polymer that is used by people daily for domestic packaging or preservation. The material by Print Pack is used for wrapping individual cheese slices for large scale production. HB1 is a trial material that has been manufactured by Phillips, and is not yet on the market. It was chosen since its oxygen and moisture permeability values are much lower as compared with the other material.

The moisture and oxygen permeability figures for these materials are illustrated in Table II. The moisture permeability values cover a range from

3.0 $g.m^{-2}.day^{-1}$ to 8 times the value, i.e: 24.0 $g.m^{-2}.day^{-1}$. The range of oxygen permeabilities is 0.5 $ml.m^{-2}.day^{-1}$ as the lowest oxygen permeability in the case of HB1 material, and the highest is 6920 $ml.m^{-2}.day^{-1}$ for DK11 material.

Table II. Permeability Values of the 4 Kinds of Packaging Materials Used

	HB1	Saran	Print-Pack	DK11
Moisture Permeability ($g.m^{-2}.day^{-1}$)	4.1075	3.0	6.51	24.0
Oxygen Permeability ($ml.m^{-2}.day^{-1}$)	0.5425	20.0	160.0	6920.0

Chemically, DK11 is composed of Styrene-Butadiene Copolymers (SBC), and its thickness is 1mil (0.025 mm). Saran technically is Polyvinylidene Chloride, and its gauge thickness is 0.8 mil (0.02 mm). The high barrier film has a 2 mil gauge thickness (0.05 mm) and it is a co-extruded PE (Poly-ethylene) and SBC (Styrene Butadiene Copolymer) film. The Print-Pack material is a 3-layer co-extruded polypropylene film of 1.2 mil (0.03 mm) thickness.

Qualitative Analysis

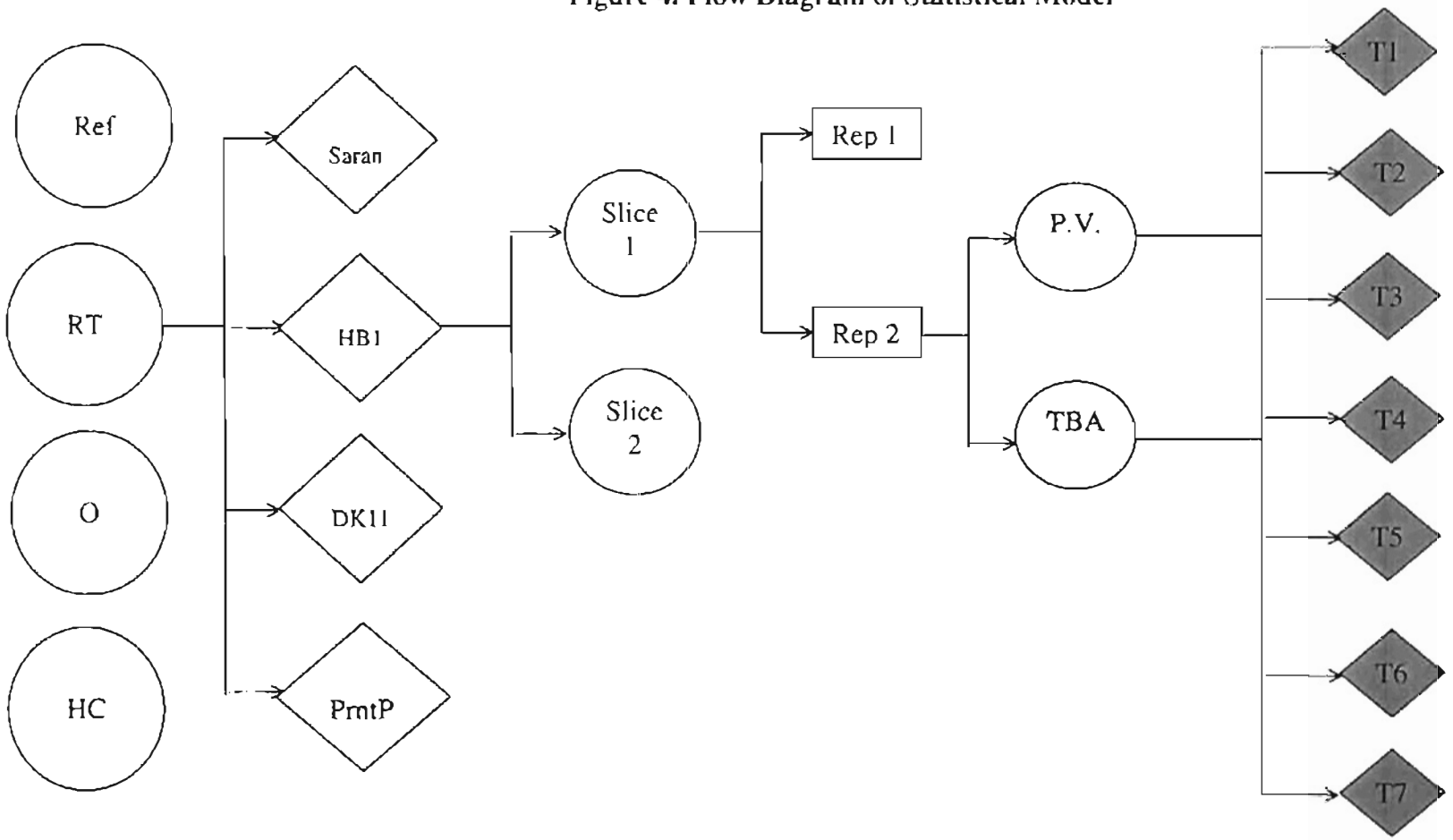
A subjective analysis of properties such as color, texture and aroma of the slices was conducted at each time step by making notes describing the specific changes in each of the slices subjected to various conditions and packaged in the four different materials. Though these tests were of a subjective nature, they provide a great deal of information about the visual and aromatic quality of the samples over time, which may help to determine the acceptability of them by consumers at various storage times.

Statistical Model

For the purpose of experimentation, two samples were drawn from each material-storage combination, and two replicates were tested from within the same slice This means that for either the Peroxide Test or the TBA Test for one point in time, there were 4 x 4 x 2 x 2 = 64 samples tested from 32 different slices, 8 each from each of the four storage conditions. Statistically, this is an experiment with a 4 x 4 x 2 factorial treatment structure in a Completely Randomized Design (CRD) with 2 repetitions at each combination of storage and material and 2 sub-samples per repetition at 7 time periods. Figure 4 illustrates a flow diagram of the statistical model.

Since there were 4 different packaging materials and 4 different storage conditions in the study that were tested at 7 times, there were 4 x 4 = 16 combinations to be studied at each time step. For each combination, two slice samples were drawn for each of the two tests, the TBA test and the Peroxide Test. Two test samples were taken from within each slice. Therefore, for one combination of storage and material, there were 2 x 2 4 replicates that were subjected to the tests. The TBA and P.V. results reported in Appendices A.1 to A.8 are the average of these 4 replicates and their standard deviations.

Figure 4. Flow Diagram of Statistical Model



Ref : Refrigeration at $4^0C \pm 1^0C$, <20%RH RT : $25^0C \pm 5^0C$, 45%RH ± 10%
O : $40^0C \pm 1^0C$, <20%RH HC : $40^0C \pm 1^0C$, 80% RH ± 2%
T1 - 1 week, T2 – 1month, T3 – 2months, T4 – 3months, T5 – 4months, T6 – 5months, T7 – 6months

Table III. Statistical Table of Inputs Used in the Analysis

Source	Degrees of Freedom (DF)
Material	3
Storage	3
Material x Storage	9
Slice within Material x Storage	16
Sample within slice	32
Time	6
Time x Material	18
Time x Storage	18
Time x Material x Storage	54
Method	1
Error	288

Assumptions Made in the Experiment

It was assumed that the sealing of all the four packaging materials was adequately efficient at the same temperature setting in the impulse sealer. It was also assumed that the temperature and humidity variations under all conditions of storage were negligible. Also, the fact that exposure to light and the intensity of light incident on the peanut butter slices could cause a part of the oxidative rancidity was not taken into account. It was assumed that all parts of one slice were equally prone to rancidity, i.e: the parts of a slice adjacent to the sealed ends were no different from areas away from the sealing joints.

CHAPTER (4)

RESULTS AND DISCUSSION

This chapter includes the results from both the qualitative analyses and the two rancidity tests : the Peroxide Test and the Thio Barbituric Acid Test. The data include results during each storage time and the statistical analysis of those results.

Peroxide Test

The results obtained from the Peroxide Test are shown in Figures 4 – 7 for each of the different packaging materials. It can be seen that the Peroxide Values (P.V.) ranged from 80 -- 350 meq/kg, i.e: the P.V. of freshly made samples was about 80meq/kg. and the maximum P.V. that most of the samples attained during their course of storage was 350 meq/kg. The time when the samples exhibited the maximum P.V. varied for each sample depending on the type of packaging material, as well as the storage condition. In general, the peroxide values seemed to increase steadily from the initial value until a maximum was reached and then the values started decreasing, which is in tune with the normal trend of peroxide formation during oxidation of fatty foods. During this process, peroxides are formed as the first by-product of fat oxidation, and then they gradually decompose into secondary by-products.

The average Coefficient of Variation (C.V.) for the Peroxide Values is 3.89%. C.V.'s ranged from 0% to 18.04% in these tests and the values are listed in Appendices A.9 – A.12.

Figure 5. Peroxide Test Results for Slices in Print Pack Material

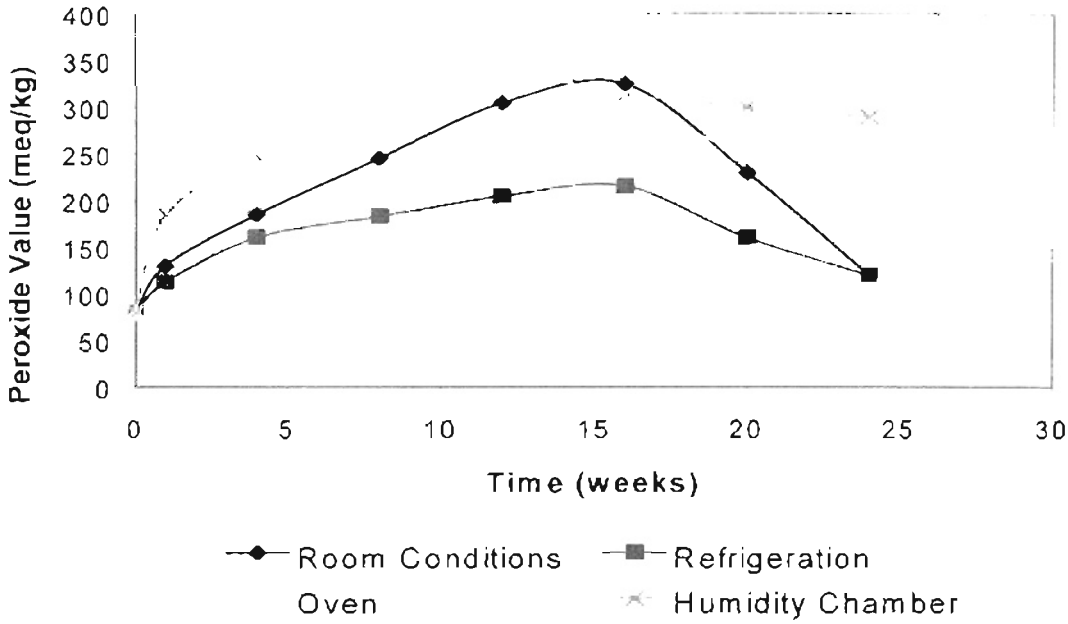


Figure 6. Peroxide Test Results for Slices in HB1 Material

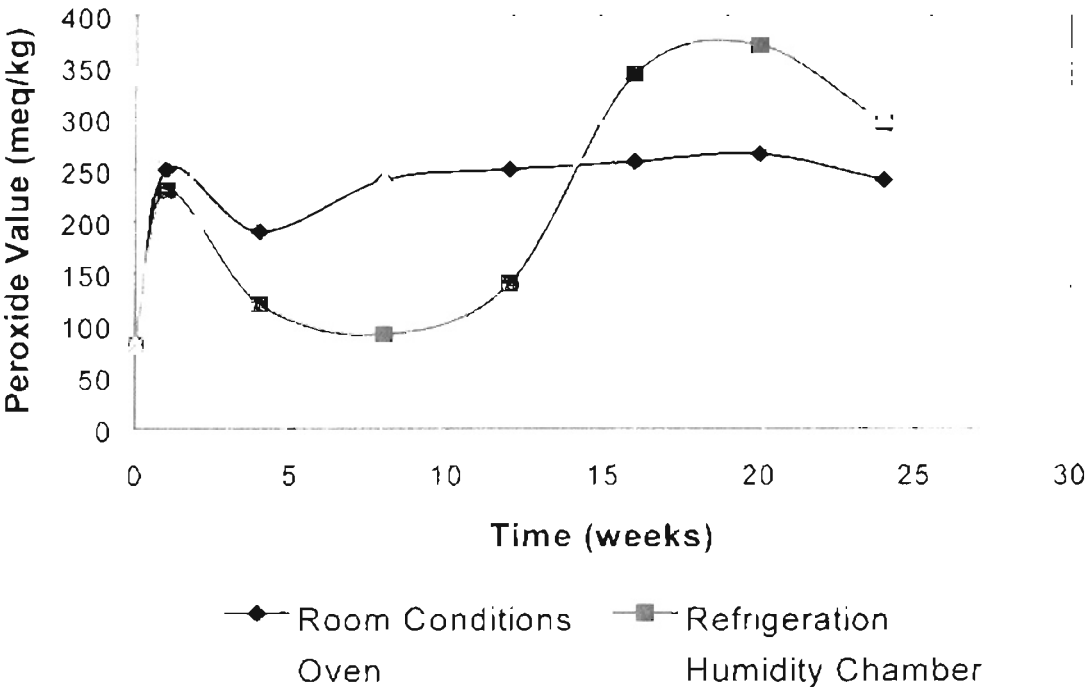


Figure 7. Peroxide Test Results for Slices in Saran Material

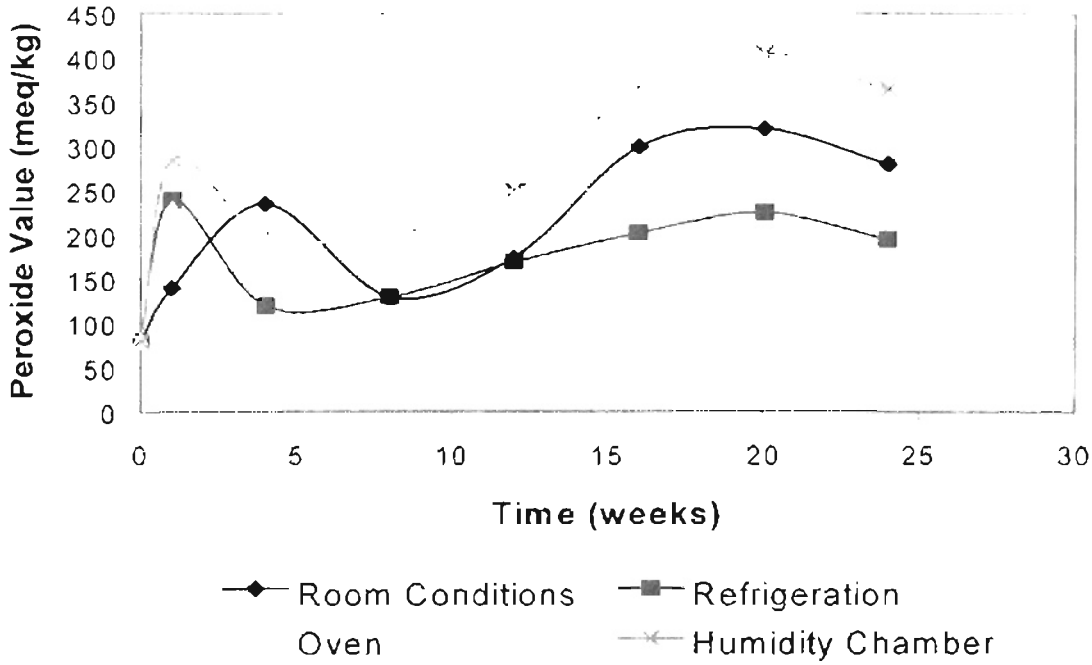
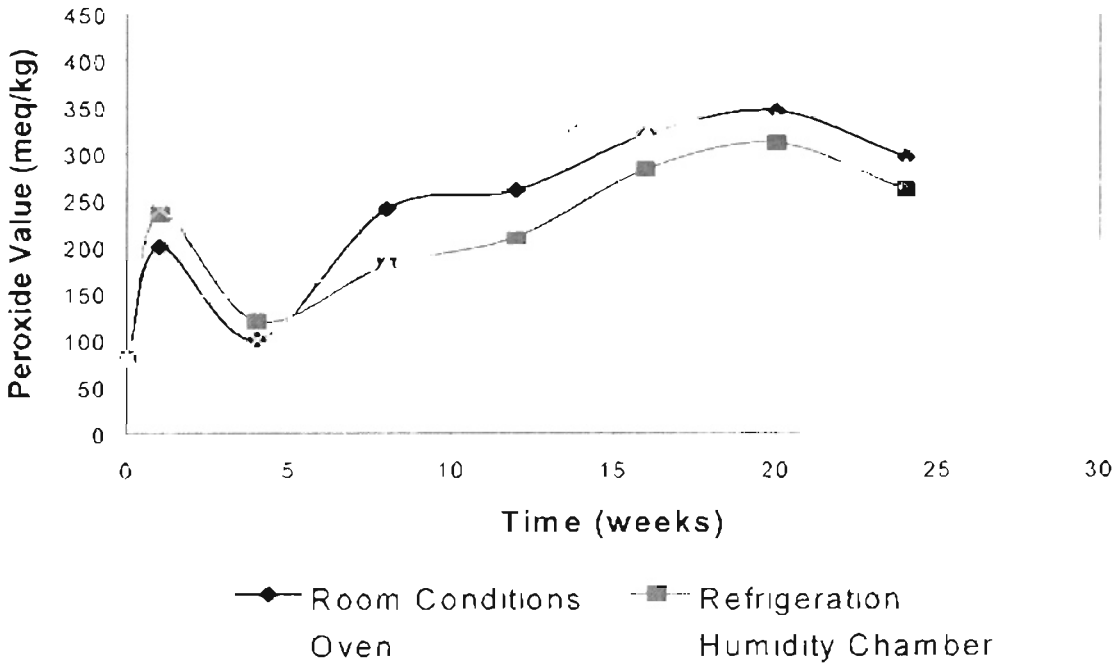


Figure 8. Peroxide Test Results for Slices in DK11 Material



Thio Barbituric Acid Assay

The results obtained from the TBA Test are shown in Figures 8 – 11 for each of the different packaging materials. It can be seen that the TBA values ranged from 0.1 to 2.2 depending on the packaging material and storage condition. For comparison, fresh beef has a TBA value between 0.1-0.2, and beef just at the onset of spoilage has a TBA value of 1.8 (Pearson, 1968). In general, the TBA values seemed to increase steadily from the initial value and continued to increase during the entire time of storage. As the TBA value gives an indirect measure of the aldehydes formed during lipid oxidation, this trend in the graphs may be explained by the fact that as the duration of storage increases, more and more aldehydes are formed within the food product. Some aldehydes are secondary or tertiary by-products of the fatty acids in the food products. The rest could be by-products of other non-fat constituents in the food material.

In general, there was a considerable amount of variability in the data and in the degree of variation from one graph to another for the same method, even if the general trends are more or less similar in most graphs. The reasons for the variabilities are not well defined, but potential reasons are difficulty in determining the end point in titration during the Peroxide Tests, and different color intensities during TBA tests. Since peanut butter has an original color which imparts a yellow color to the reaction mixture during the Peroxide Tests, it becomes exceedingly difficult to judge the precise end point during titration, as the end point is determined by color change of yellow potassium iodide to colorless. The solution almost never becomes colorless due to the original color of peanut butter. During TBA tests, all the samples are centrifuged together and then their color intensities are read one at a time. Since the samples whose intensities are read

towards the end are allowed to stand for a few hours longer than the rest, their color intensities change visually. This could cause the spectrophotometer readings to be different than what they would have been if all the samples were measured for their color intensities at the same time. These could be some reasons why the TBA and Peroxide Values exhibit a wide range of variability. A sudden increase in the TBA values for all materials was observed around the one month storage period. This could indicate that the reaction mixture was allowed to stand for too long before taking the spectrophotometer readings. The average Coefficient of Variation of the TBA values was 6.215, with C.V.'s ranging from 0.153 to 54.562. All C.V.'s are listed in Appendices A9 – A.12.

Effect of Packaging Material

Each packaging material displayed different characteristics with respect to influencing rancidity, as well as other properties like ease of sealing and its peelability from peanut butter slices. Since the sealer used was a heat-impulse sealer, the factor that influenced the sealing quality was mainly the temperature. Saran could seal at low temperature as compared to the others, and the Print Pack material required higher degrees of heating as compared to the others. Also, DK11 exhibited minimum adherence to the peanut butter slices, followed by the Print Pack material, HB1 and Saran. However, these observations with respect to the sealing quality and peelability of the packaging materials were very subjective in nature.

The Print Pack packaging material displayed the most consistent behavior with respect to the rancidity patterns of peanut butter slices. From both the TBA as well as the Peroxide Test graphs of Print-Pack (Fig 4 and 8), it can be seen that the samples packed with this material began to degenerate after 16 weeks (4 months) of storage under

room conditions. There was a relatively small change with respect to the P.V. and TBA values of the slices wrapped with the Print-Pack material and stored under refrigerated conditions, even after 6 months, when compared with the slices packaged in the other materials.

The high barrier material, HB1, also had relatively good performance with respect to the storability of peanut butter slices. Under refrigerated conditions, there was no oxidation until about 16-18 weeks, as is evident in both the TBA and the Peroxide Test results (Fig. 5 and 9). Under room conditions, rancidity developed at around 12 weeks.

Figure 9. TBA Results for Slices in Print Pack Material

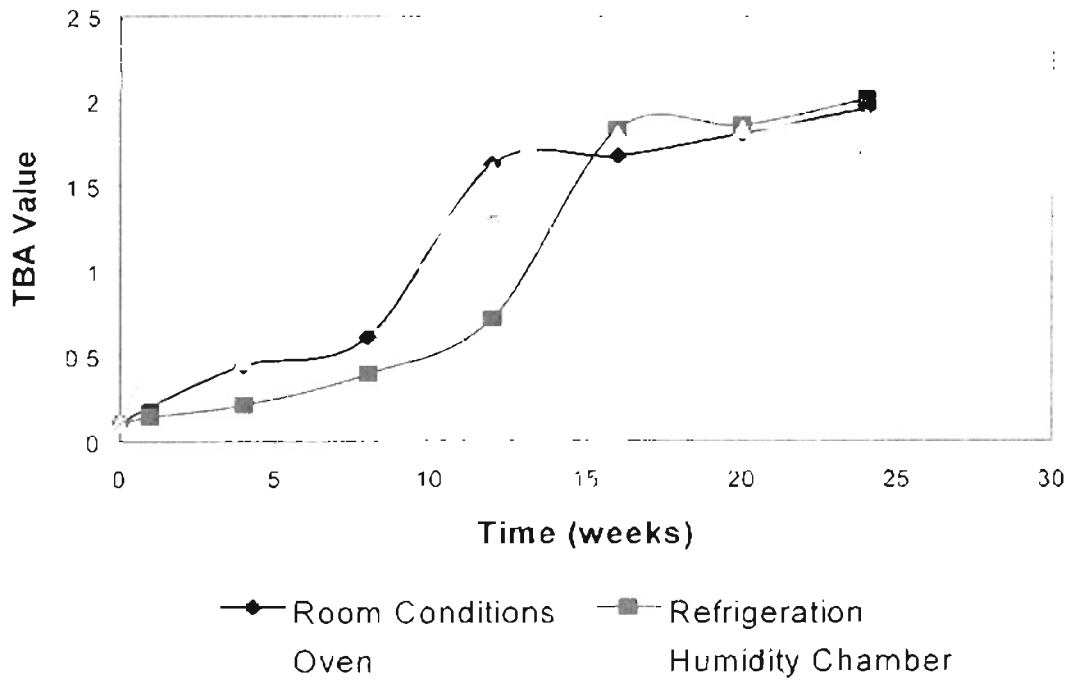


Figure 10. TBA Results for Slices in HB1 Material

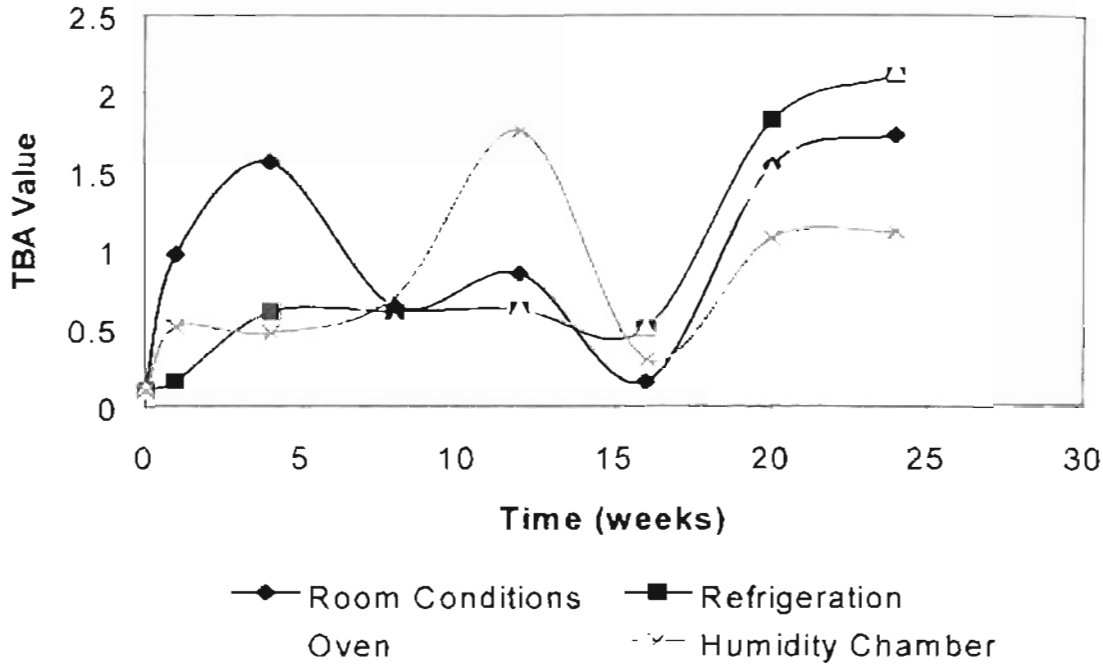


Figure 11. TBA Results for Slices in Saran Material

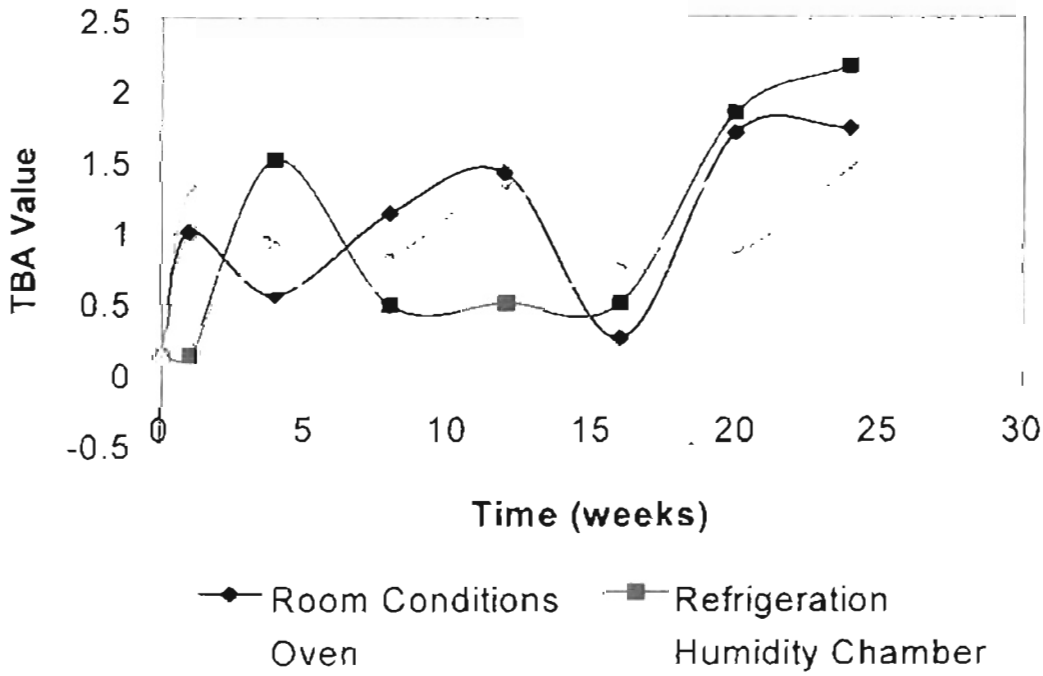
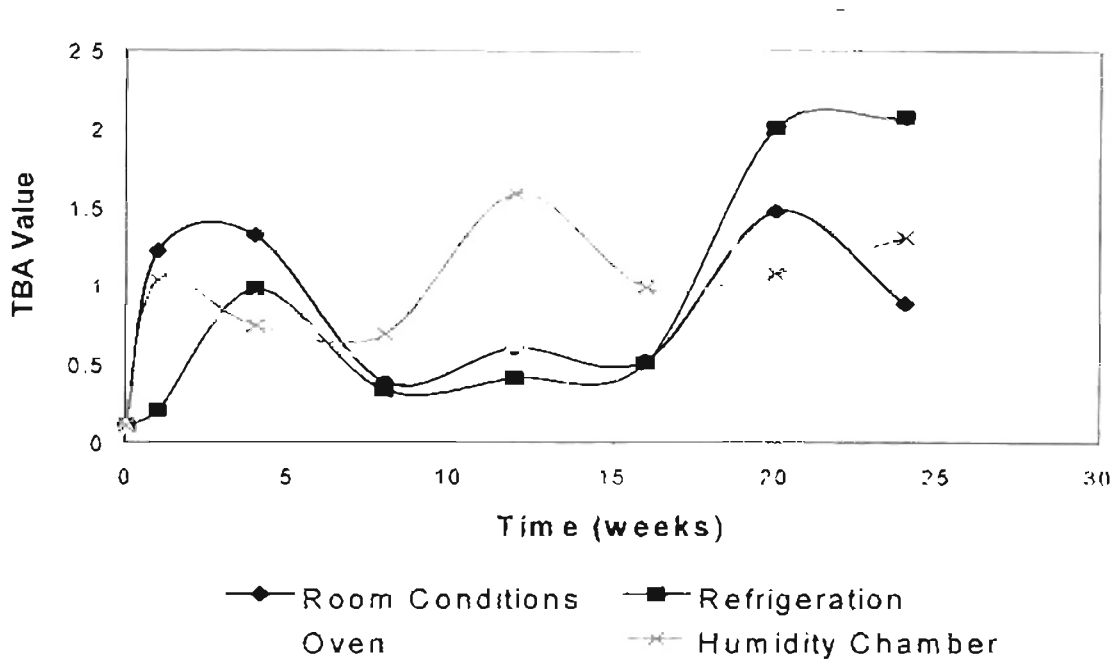


Figure 12. TBA Results for Slices in DK11 Material



Saran wrap performed fairly well until about 8-10 weeks under refrigerated and room conditions. In the oven as well as humidity chamber, the slices exhibited considerable rancidity in less than 6 weeks (Fig. 6 and 10). There was a little seepage of oil in Saran under elevated temperatures and humidity, i.e: during storage in the oven at 40^{0}C, and in the humidity chamber at 40^{0}C and 80% relative humidity.

DK11 was found to be the most unsuitable material for packaging of the peanut butter slices. There was a lot of visible oil seepage through the DK11 packaging material under all conditions of storage, primarily after 3 months of storage. Even under refrigerated conditions, there appeared to be an adequate amount of rancidity in less than 8 weeks (Fig.7 and 11). These results can be expected in light of the oxygen permeability figures of the packaging materials illustrated in Table II. Overall, the slices wrapped in Print-Pack and HB1 (the high barrier material) exhibited better shelf-life properties than Saran and DK11.

Effect of Storage Conditions

Refrigerated storage conditions were ideal for storage of peanut butter slices. No significant change occurs in the slices stored under refrigeration even after 6 months of storage, in three of the four packaging materials (all except DK11). There was rapid aroma loss in the peanut butter slices after about 4 months of storage, except those stored in the refrigerator. The aroma loss was detected using visual and odor sensing, and hence the quality of this observation is very subjective. The original color of the slices is also retained to the highest degree in the refrigerated slices, as shown in Figure 16, which shows the slices packaged in all four kinds of packing materials that had been refrigerated for 6 months.

Under room conditions, Print Pack performs the best with no significant rancidity development before 14-15 weeks of storage time. Around that time, the peroxide values start dropping for the Print Pack material, but they still continue to increase for the other materials (Figure 12, App.B.1), and the TBA values for the Print Pack material stabilize at that time, while they are still fluctuating for the rest of the materials. For all the other packaging materials, considerable rancidity develops in less than 10 weeks of storage. The distinctive aroma of peanut butter disappears after 5 months of storage at room conditions irrespective of the packaging material used. After 6 months of storage at room conditions, the slices packed in all materials lose their initial rich color and become pale looking (Fig. 17).

When stored in the oven at 40^0C, the slices packed in the Print-Pack material last for about 10 weeks. The remaining samples undergo rancidity changes within 5-6 weeks of storage (Figures 13 and 14). This is concluded because the TBA

values are still fluctuating after 10 weeks for all other materials and peroxide values have not reached a well defined peak value even after 10 weeks for all materials except the Print Pack material. The aroma vanishes after 3 months of storage in the oven for all materials.

For the samples stored in the humidity chamber, a very distinct characteristic that is observed is the high degree of oil seepage through all the barrier materials except Print Pack, as compared to the other storage conditions. The oxidative shelf-life of the peanut butter slices in the humidity chamber is close to that of the slices in the oven, i.e: 10-12 weeks when packed in Print-Pack, and 5-6 weeks in all other packaging materials. It was observed that the slices in DK11 develop a dark color and brittle texture after 4 months of storage in the humidity chamber. Also, there was a great deal of fungal growth around the slices that were packed in DK11 and stored in the humidity chamber after 6 months of storage, which is shown in Figure 15.

Figure 13. Peroxide Test Results Under Room Conditions

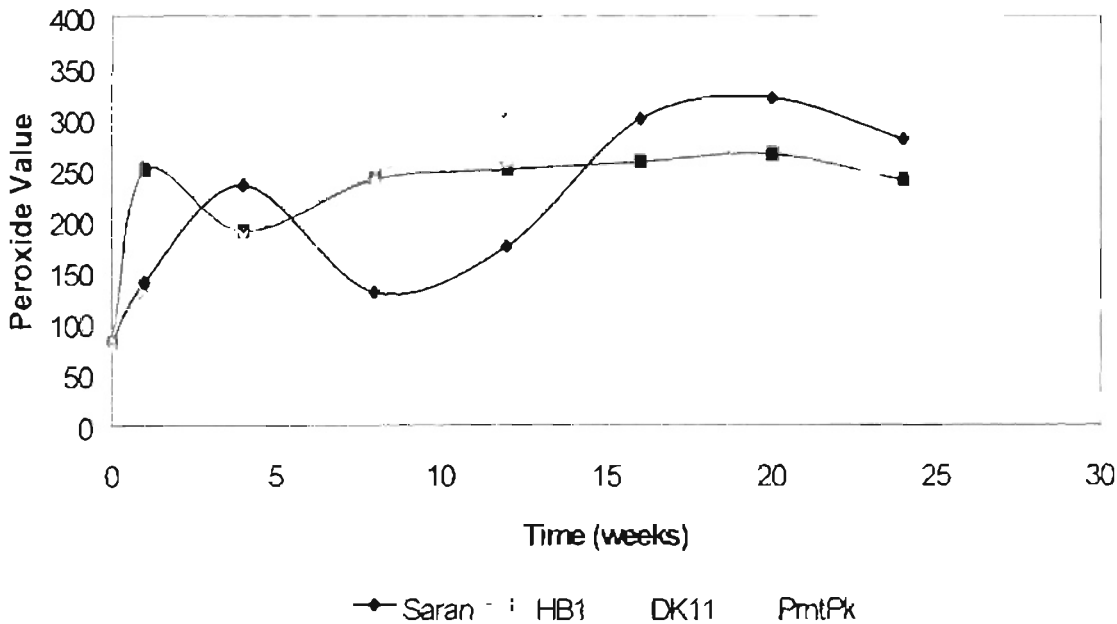


Figure 14. Peroxide Test Results Under Oven Conditions

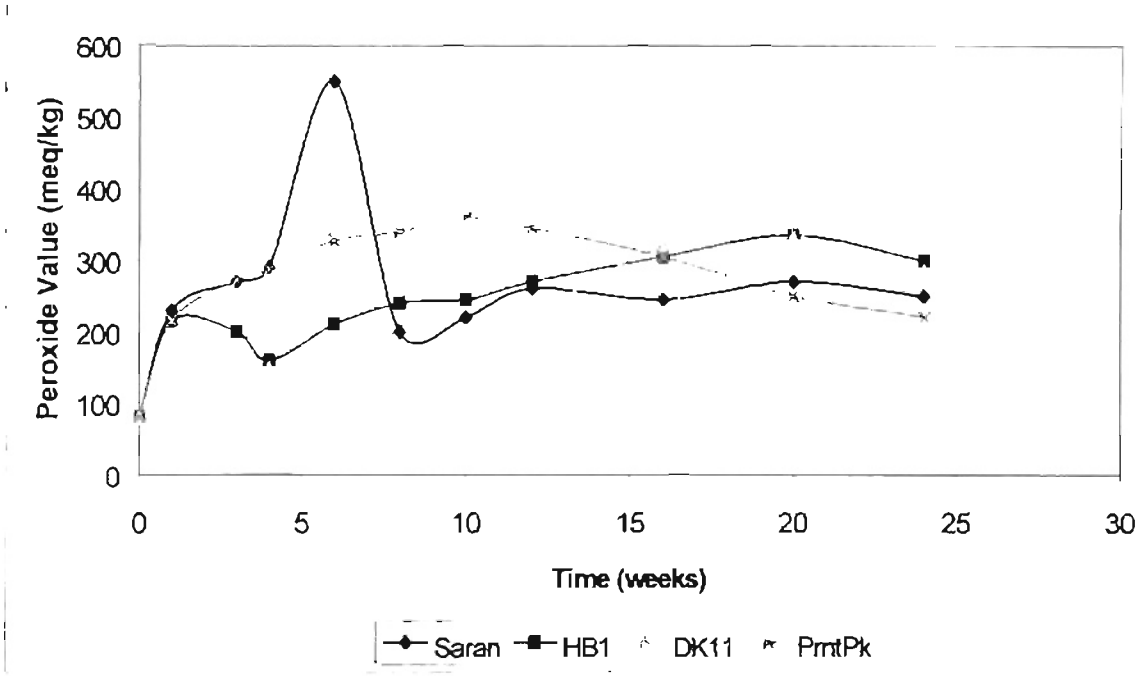
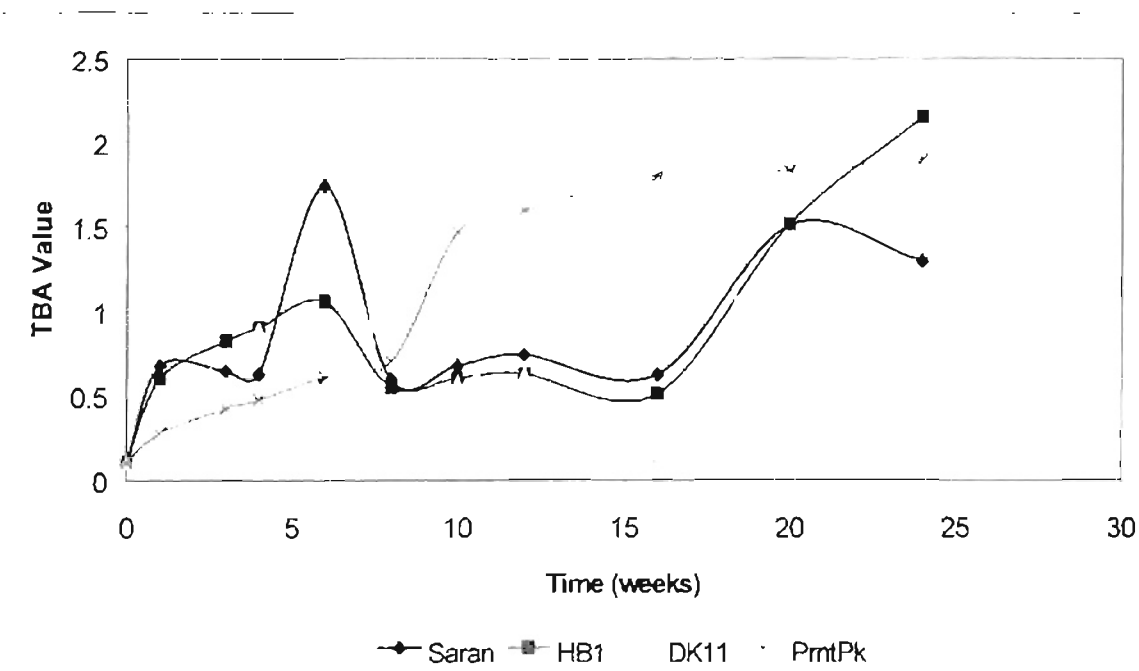


Figure 15. TBA Results Under Oven Conditions



The degree of rancidity was higher for slices stored in the oven than the humidity chamber. This observation might be used to conclude that moisture doesn't play a very significant role in acceleration of oxidative rancidity of peanut butter slices.

Appendices A.1 through A.8 list the numerical results of the TBA and Peroxide tests done on each peanut butter slice over the six month storage period. Appendices B.1 through B.5 illustrate some of those results in the form of graphs.

Figure 16. Peanut Butter Slices After 6 Months of Storage in Humidity Chamber

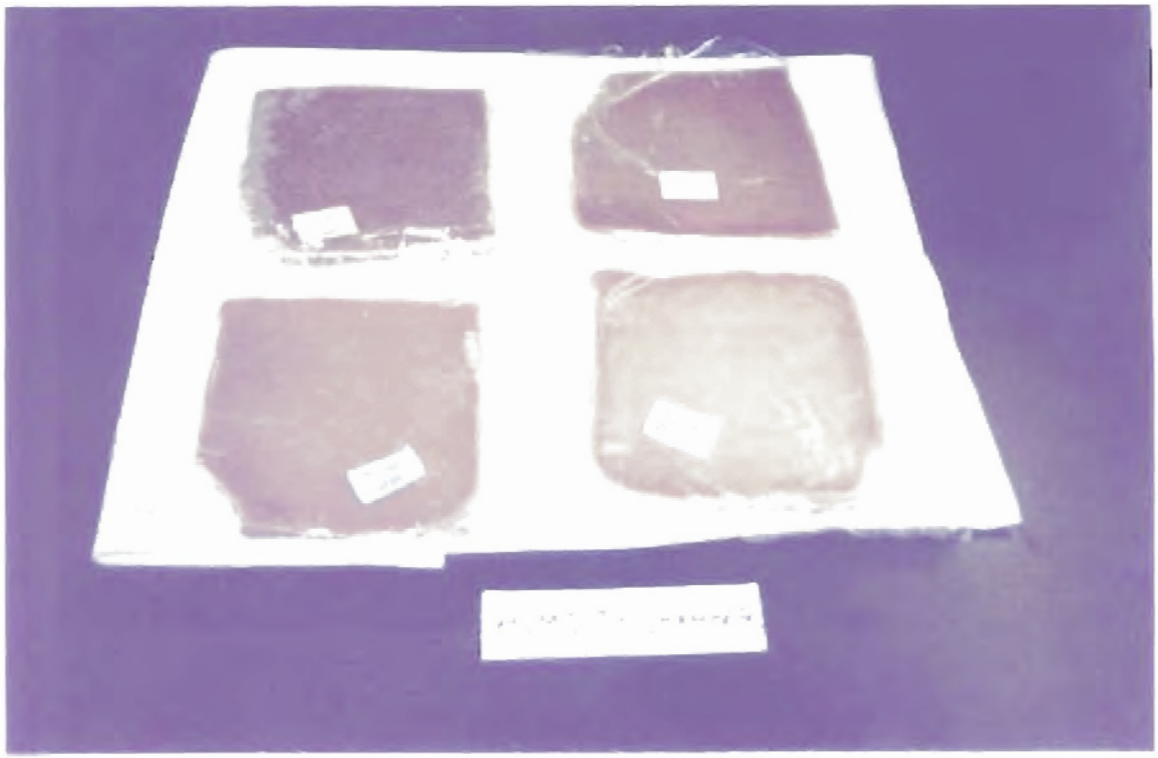


Figure 17. Peanut Butter Slices After 6 Months of Storage in Refrigerator

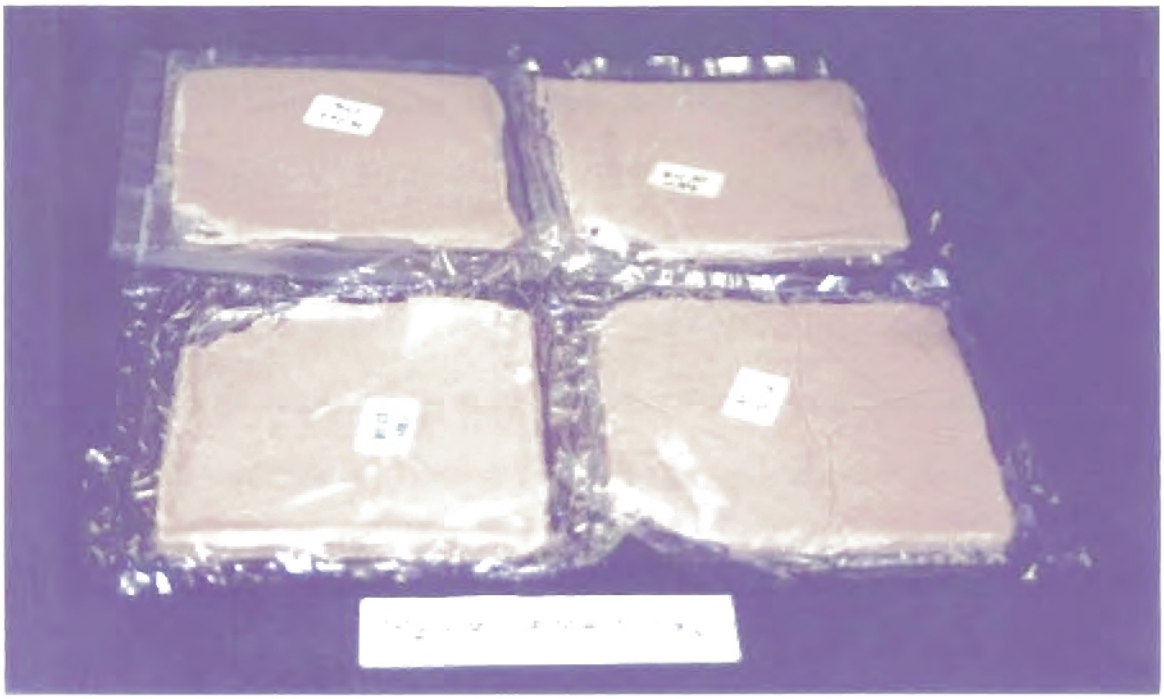
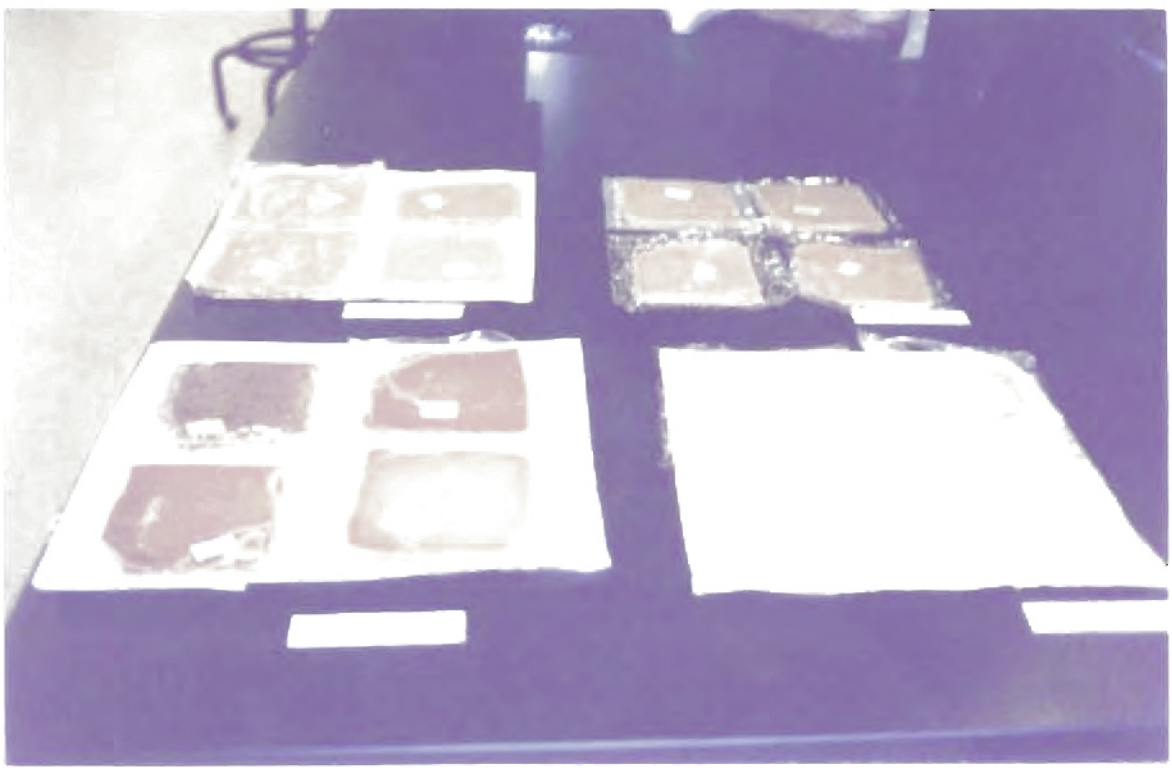


Figure 18. Samples Under all 4 Storage Conditions After 6 Months



Variation in Qualitative Properties

The physical properties of the peanut butter slices that were qualitatively evaluated, such as aroma, color and texture, showed a wide range of variation under different conditions of storage. These physical properties seemed to change more with respect to the storage condition and were less influenced by the kind of packaging material used. Most slices underwent a color change from original color to very dark shades of brown during storage in the humidity chamber. In addition, the slices developed a brittle texture during storage in the humidity chamber. Also, under room condition, most slices turned pale. Table IV gives an overview of the effect of various storage conditions on the change in some physical properties of peanut butter slices for different packaging materials.

Table IV. Qualitative Property Changes During Storage

Material	Color	Texture	Aroma
Print Pack	Darkens in H.C. after 5 months. Turns pale in R.C. after 5 months.	Turns brittle in H.C. after 5 months. Sticky in O after 4 months.	Retained under Ref. and R.C. Lost after 4 months in oven and H.C.
HB1	Darkens in H.C. after 4 months. turns pale in R.C. after 5 months.	Turns brittle in H.C. after 5 months.	Retained under all conditions until 6 months.
Saran	Turns pale under R.C. after 3 months. Darkens in H.C. after 4 months.	Sticky under oven conditions after 3 months. Brittle after 4 months in H.C.	Retained until 4 months under Ref. Lost after 3 months under all other conditions.
DK11	Darkens after 4 months in H.C. Turns pale under R.C. after 3 months	Brittle after 3 months in H.C.	Lost after 4 months under all conditions.

Note : The storage conditions are abbreviated as
R.C. - Room Conditions (25^0C ± 5°C, 45%RH ± 10%)
Ref. - Refrigeration (4^0C ± 1^0C, <20%RH)
O - Oven (40^0C ± 1^0C, <20%RH)
H.C.- Humidity Chamber (40^0C ± 1^0C, 80% RH ± 2%)

Temperature Effects on Rancidity

From the experiments, it was observed that storage temperature had a more pronounced effect on the development of rancidity than the humidity to which the peanut butter slices were subjected. A temperature range of 5^0C-40^0C was covered under the 4 different storage conditions that were selected. Refrigerated storage was at 5^0C, room temperature was at 25^0C and the temperatures both in the oven and the humidity chamber were at 40^0C. In Figures 5-12, the time at which a Peroxide curve reached its peak and the time at which the slope of the TBA curve abruptly stopped increasing after reaching a high value were estimated. The estimated values for each of the experimental conditions (Tables V and VI) were plotted as a function of storage temperature which are shown in Figures 19 to 22.

It can be clearly seen that the slices stored under refrigerated conditions take longer to show signs of rancidity for both the TBA and Peroxide Values. The relationship between the temperature and the time when the slices start exhibiting rancidity is linear, as is shown by the regression equations in the graphs. All the trend lines have a negative slope, as the peak time for rancidity development is smaller for higher temperatures, and the R^2 values range from 0.68 to 0.99. In Figures 19 and 20, the trend line for Prink Pack has the smallest negative slope of –0.11, as compared to the slopes of the other trend lines which are all approximately –0.21. This may be used to conclude that Print Pack is less sensitive to temperature differences with respect to rancidity.

Table V. Time (weeks) to Onset of Rancidity from peak Peroxide Values

Storage Temperature (^{0}C)	Time to Onset of Rancidity (weeks)			
	PrnkPk	HB1	Saran	DK11
5	16	18	20	16
25	16	12	16	16
40	12	10	12	8

Table VI. Time (weeks) to Onset of Rancidity from TBA Values

Storage Temperature (^{0}C)	Time to Onset of Rancidity (weeks)			
	PrnkPk	HB1	Saran	DK11
5	16	20	16	16
25	12	20	12	16
40	12	15	12	12

Figure 19. Peak Rancidity Time vs Temperature for Peroxide Test – PmtPk & HB1

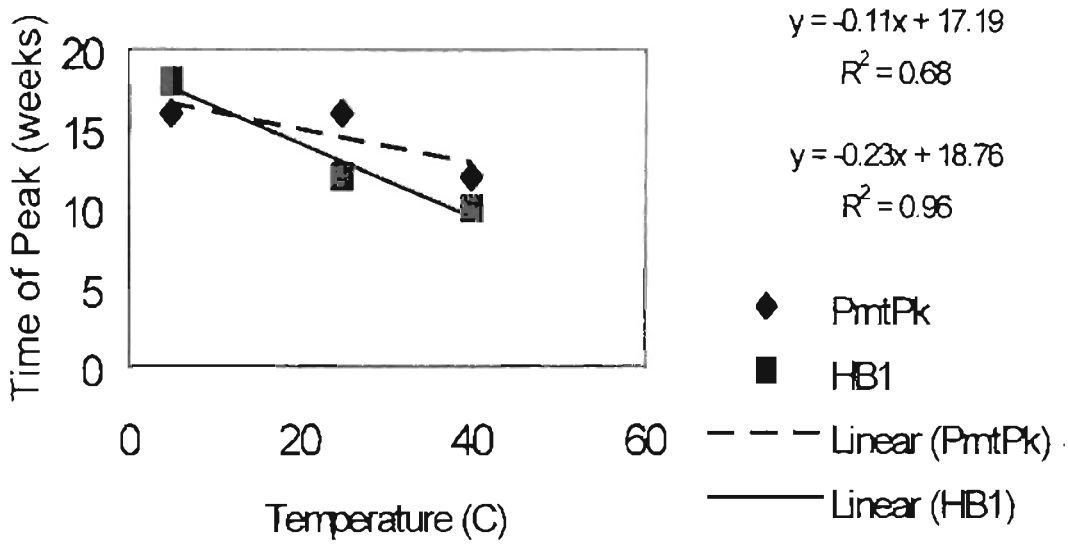


Figure 20. Peak Rancidity Time vs Temperature for Peroxide Test - Saran & DK11

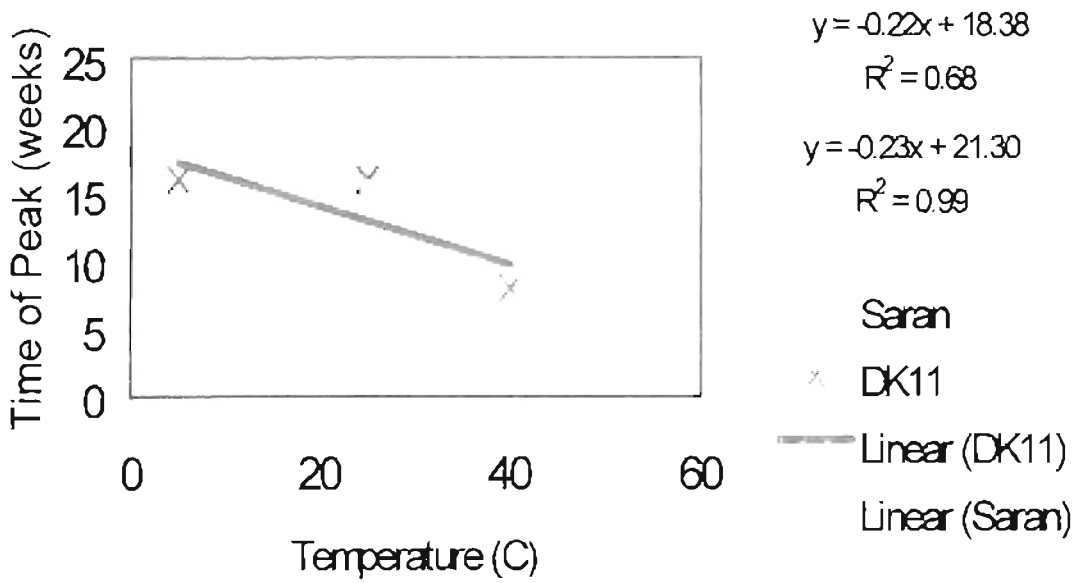


Figure 21. Time of Onset of Rancidity vs Temperature for TBA Test – PrntPk & HB1

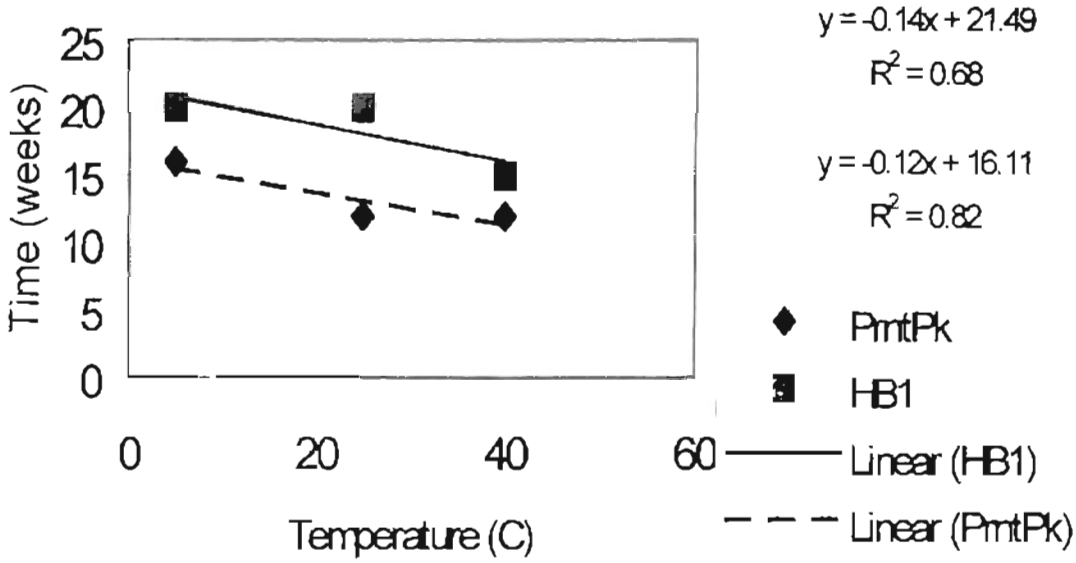
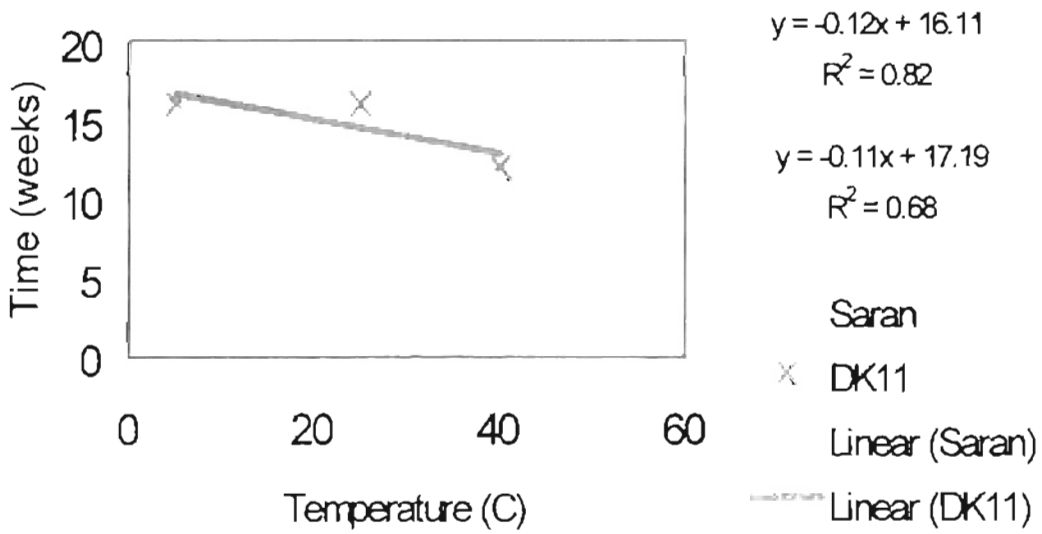


Figure 22. Time of Onset of Rancidity vs Temperature for TBA Test – Saran & DK11



The rancidity development at each of the time steps can be seen in Figures 23 & 24. These figures show the actual rancidity values with increasing temperature at each of the time steps for both the TBA and Peroxide tests for two different packaging materials. The remaining graphs are shown in appendices A.6-A.11. These graphs confirm that the temperature effects on rancidity development are significant. During the early part of storage, rancidity values increase with increasing temperature.

An interesting phenomenon can be observed in some of the graphs at later time periods. The rancidity increases with increasing temperature during the first part of the storage period (until the peak is reached), and decreases with increasing temperature for the second half of the storage period. As it is not feasible that the already rancid samples can get any better as time progresses, this observation might be explained in terms of breakdown of those rancidity components that were evaluated in each test into further by-products. Also, it might be concluded that the higher the temperature, the higher is the rate of breakdown of the rancidity by-products with progressing time. However, this trend is not so evident in the P.V. graphs for DK11, Saran and Print Pack materials. For these packaging materials, the peroxide values seem to increase with increasing temperature at every time step. This indicates that the peanut butter fats continue to get oxidized and form peroxides constantly even after 6 months. This might be used to conclude that peroxides are more prominent byproducts of oxidation as compared to the aldehydes.

Figure 23. Temperature Effects on Peroxide Values for HB1 Packaging Material

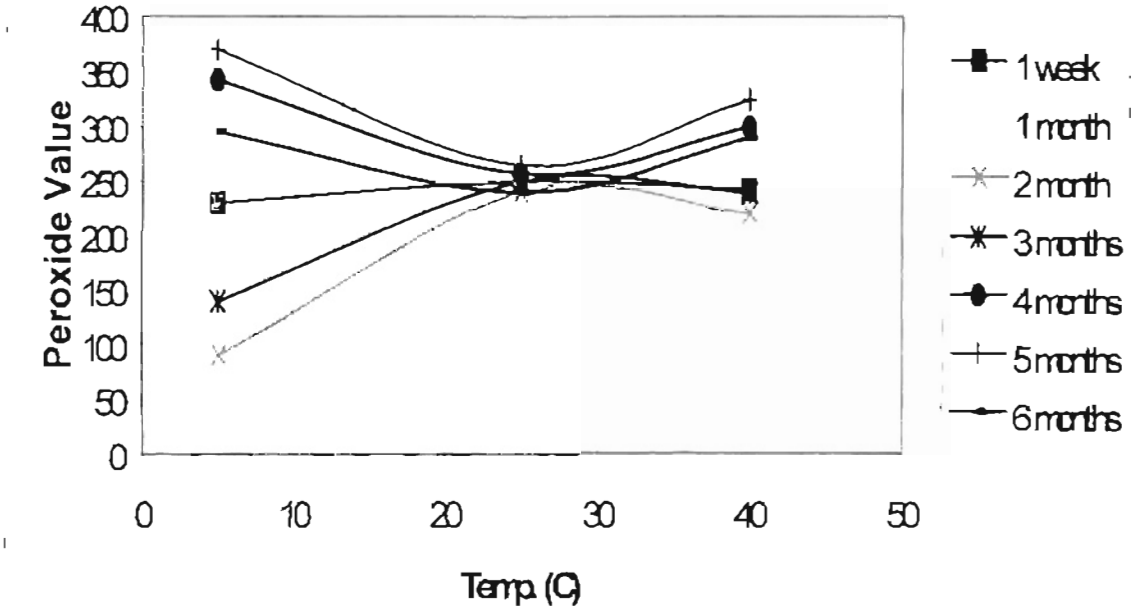
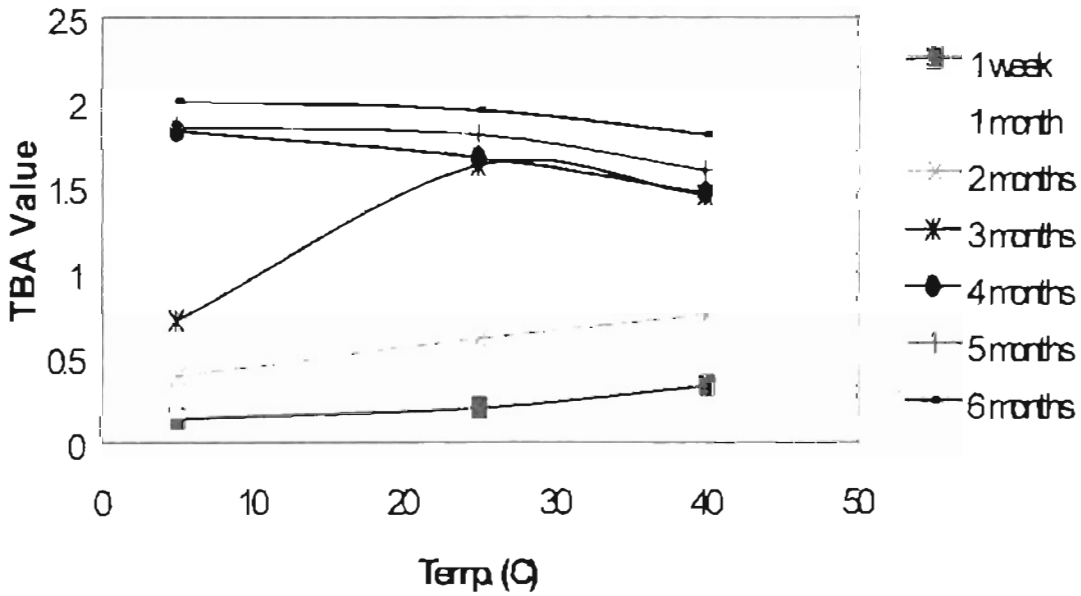


Figure 24. Temperature Effects on TBA Results for Print Pack Material



Statistical Analysis

The statistical model involves two test methods, four packaging materials and four storage conditions with two repetitions x two sub-samples per repetition and seven time periods. The treatment structure is a 2x4x4 factorial, and the design structure is a Completely Randomized Design (CRD).

The statistical analysis of the data and the interactions between all the variables in the experimental layout is illustrated in Appendix C. The analysis was performed after converting collected data to log(%Control) + 1, which means that all raw data was transformed into the percentage of initial data (at time '0') which it represented. The data that was used to plot the graphs shown in Appendix C.3 are that of the least square means of the Peroxide or TBA values against time. Same plotting symbols indicate same material.

From the analysis as well as from the graphs, it is apparent that there is significant interaction between method, material, storage condition and time ($p<0.0001$). Also, the packaging materials are significantly different from each other, and so are the storage conditions. All estimations were done at 99.99% confidence interval (page 83, Type 3 Tests of Fixed Effects).

The following observations were made with respect to each of the rancidity tests :

In the peroxide test, samples stored under refrigerated conditions have a significant material effect across all times. DK11 has no storage effect after 2 months of storage, which could mean that the samples, when packed in DK11, undergo maximum deterioration within 2 months. From results of Thio Barbituric Acid test, refrigerated

samples show no material effect after 5 months. The storage conditions affect TBA results for each material, except Print-Pack at 6 months and HB1 at 2 months.

From the analysis of interaction between storage and material for the Peroxide Test, it may be inferred that the variations among different material and storage combinations are significantly different ($F(9,16) = 5.47$, $p = 0.0016$). Under conditions in the humidity chamber there is no significant difference between the performance of all 4 packaging materials ($F(3,16) = 1.38$, $p = 0.2846$). This means that all packaging materials perform equally poor under high temperature, high humidity conditions. Also, there is no significant storage condition effect on slices packaged with HB1 material ($F(3,16) = 2.27$, $p = 0.1195$). Therefore, samples packaged in HB1 may be less prone to degradation due to changes in atmospheric conditions for a short period of storage. This could be due to the HB1 material having the lowest oxygen permeability of the four materials (Table II).

The Print Pack material exhibits significantly different responses between storage under high temperature/high humidity and that under low temperature/low humidity, indicating that temperature has a considerable effect on this packaging material. It might be a suitable material for long term storage under refrigerated conditions or under room temperature, but is not optimal at high temperature (page 102).

Saran has a significant storage effect ($F(3,16)$ 6.47, p 0.0045). However, the mean response under room conditions is very much lower than the mean under other conditions, which suggests that Saran performs best at moderate temperatures (page 99).

The results from the two test methods varied significantly for nearly all material x storage x time combinations at the 95% level, except in the case of DK11 at room conditions in the 2nd and 4th months, HB1 in refrigeration in 4th month, Print-Pack in oven, room conditions and refrigeration in the first week and Print-Pack in refrigeration after 1st month. This could be used to conclude that one test might not replace another, and it is advisable to conduct both, the Peroxide Test, as well as the TBA Test on peanut butter slices.

CHAPTER (5)

CONCLUSIONS

The rancidity studies on peanut butter slices provide some insight regarding the most suitable kinds of packaging and storage conditions for this product. The conclusions drawn from this study are as follows :

1. Rancidity begins after 1-2 months of storage under room conditions for all packaging materials except Print Pack. in which the peanut butter slices last for about 15 weeks before showing signs of rancidity.
2. The high barrier packaging material, HB1, and the Print Pack film performed the best under all conditions of storage. The HB1 material may be better for short term storage owing to its very high oxygen and moisture permeability values.
3. DK11 is the least suitable of the packaging materials tested.
4. Peanut butter slices exhibit good shelf-life properties of up to 6 months under refrigeration for all kinds of packaging materials used, except DK11, which provides very low oxygen barrier. Both TBA and Peroxide Values were much smaller for refrigerated samples than samples at higher temperatures. Peanut butter slices retained their normal aroma and color even after 6 months of storage in the refrigerator for all packaging materials except DK11.
5. Storage temperature apparently has a greater effect on the rancidity of peanut butter slices than humidity. This was concluded based on the fact that under the same conditions of packaging and storage time, the TBA results and Peroxide Values were a little higher under oven conditions than in the humidity chamber in most cases.

6. Storage temperature and humidity affects the color of peanut butter slices significantly. High temperature and humidity cause the slices to turn dark and brittle, and storage under room conditions causes the color of the slices to go very pale.
7. Temperature plays a vital role in accelerating rancidity of peanut butter slices. For smaller storage periods, rancidity in terms of peroxide value and aldehyde generation increases rapidly with rise in temperature.
8. The Print Pack material exhibits the highest efficiency under varying temperatures.
9. Peroxide Test is better than TBA test for rancidity analysis, as shown by the lower Coefficient of Variation.

CHAPTER (6)

RECOMMENDATIONS FOR FUTURE WORK

Studies should be conducted on the release characteristics of the various kinds of packaging materials. Release characteristics refer to the ease with which the peanut butter slices can be separated from the packaging film, which will depend on the cohesive and adhesive forces between the material and peanut butter.

Microbial tests and shelf-life studies with respect to microbial spoilage should be conducted to substantiate or complement the tests for oxidative stability. Organoleptic tests must be carried out, i.e. the slices must be subjected to a sensory panel in order to assess consumer acceptance of the product after certain periods of storage.

Studies must be conducted to improve upon the laboratory scale packaging of individual peanut butter slices.

Other kinds of rancidity tests should be carried out on the peanut butter slices to supplement the Peroxide Test and Thio-Barbituric Assay. Also, the validity of these two tests used on peanut butter must be ascertained.

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APPENDICES

APPENDIX A.1

TBA Results for Room Conditions

Time (weeks)	TBA results for Saran		TBA results for HB1		TBA results for DK11		TBA results for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	0.102	5.77E-4	0.102	8.08E-3	0.102	5.77E-4	0.102	5.77E-4
1	0.988	2.56E-2	0.976	2.63E-2	1.2205	6.59E-2	0.201	3.26E-2
4	0.542	2.49E-2	1.564	5.18E-2	1.32	3.41E-1	0.443	2.49E-2
8	1.119	1.71E-3	0.642	6.86E-3	0.385	3.67E-2	0.609	1.14E-2
12	1.405	2.28E-2	0.854	3.08E-2	0.601	1.76E-2	1.628	1.59E-2
16	0.24	5.45E-2	0.16	8.73E-2	0.52	7.89E-2	1.677	1.22E-1
20	1.69	8.19E-2	1.543	3.62E-2	1.475	3.8E-2	1.81	1.69E-2
24	1.725	1.15E-1	1.744	1.54E-1	0.884	1.67E-1	1.955	1.43E-1

APPENDIX A.2

TBA Results for Refrigeration

Time (weeks)	TBA results for Saran		TBA results for HB1		TBA results for DK11		TBA results for PrntPk	
	μ	σ	μ	σ	μ	σ	μ	σ
0	0.102	8.08E-3	0.102	7.51E-3	0.102	8.08E-3	0.102	7.51E-3
1	0.122	9.29E-3	0.163	1.05E-2	0.203	6.7E-3	0.143	3.65E-3
4	1.5	5.16E-2	0.604	1.78E-2	0.978	2.47E-2	0.211	3.77E-2
8	0.471	3.8E-2	0.609	9.91E-3	0.332	3.15E-2	0.39	4.24E-3
12	0.479	1.51E-2	0.622	7.53E-3	0.409	7.14E-3	0.722	1.36E-2
16	0.489	2.92E-2	0.5	1.07E-1	0.505	8.53E-2	1.835	1.73E-2
20	1.832	7.85E-2	1.838	1.21E-1	2.006	7.92E-2	1.858	5.67E-2
24	2.164	3.01E-1	2.124	2.31E-1	2.072	2.2E-1	2.011	1.21E-1

APPENDIX A.3

TBA Results for Oven Temperature

Time (weeks)	TBA results for Saran		TBA results for HB1		TBA results for DK11		TBA results for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	0.102	7.51E-3	0.102	7.51E-3	0.102	5.77E-4	0.102	8.08E-3
1	0.675	2.32E-2	0.6	7.55E-3	0.408	8.73E-3	0.275	2.38E-2
3	0.642	2.87E-2	0.822	2.32E-2	0.734	4.21E-2	0.419	2.38E-2
4	0.623	3.95E-3	0.9	9.61E-3	0.9	1.02E-2	0.468	3.94E-2
6	1.739	1.96E-2	1.054	5.00E-3	2.099	1.01E-1	0.605	8.42E-3
8	0.593	2.87E-2	0.546	7.07E-3	0.473	5.94E-3	0.7	5.94E-3
10	0.676	5.27E-2	0.603	3.62E-2	0.579	7.3E-2	1.46	6.44E-3
12	0.74	1.02E-2	0.625	2.26E-2	0.628	1.54E-1	1.593	6.9E-2
16	0.622	5.52E-2	0.509	6.27E-2	0.13	3.93E-2	1.806	3.24E-2
20	1.509	4.23E-2	1.516	1.21E-2	1.696	5.42E-2	1.832	4.85E-2
24	1.295	2.47E-1	2.149	2.94E-1	1.579	3.87E-2	1.9	2.42E-2

APPENDIX A.4

TBA Results for Conditions in Humidity Chamber

Time (weeks)	TBA results for Saran		TBA results for HB1		TBA results for DK11		TBA results for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	0.102	5.77E-4	0.102	8.08E-3	0.102	7.51E-3	0.102	8.08E-3
1	1.282	1.33E-2	0.508	2.74E-2	1.0335	3.38E-2	0.389	3.78E-2
3	0.974	1.87E-2	0.494	2.49E-2	0.881	3.99E-2	0.577	3.78E-2
4	0.914	2.48E-2	0.47	2.23E-2	0.744	1.5E-2	0.612	6.04E-2
6	1.192	1.36E-2	1.661	3.05E-2	1.682	1.06E-1	0.707	6.58E-3
8	0.827	1.28E-2	0.707	1.93E-2	0.694	2.41E-2	0.797	2.28E-2
10	0.928	4.4E-2	0.7378	6.99E-3	0.732	1.67E-2	1.655	5.43E-3
12	1.3	3.26E-2	1.767	3.81E-2	1.587	3.61E-2	1.314	1.62E-1
16	0.758	8.67E-2	0.302	4.9E-2	0.994	1.12E-1	1.129	1.46E-1
20	0.836	6.08E-2	1.072	3.1E-2	1.076	3.06E-2	1.371	4.09E-2
24	1.44	1.09E-1	1.113	5.9E-2	1.308	6.94E-2	1.724	2.47E-2

APPENDIX A.5

Peroxide Values for Room Conditions

Time (weeks)	P.V for Saran		P.V. for HB1		P.V. for DK11		P.V.*** for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	80	14.43	80	2.89	80	11.55	80	11.55
1	140	5.00	250	9.57	200	15.00	130	5.00
4	235	10.00	190	5.00	100	9.57	185	17.32
8	130	8.16	240	0.00	240	5.00	245	5.77
12	175	5.77	250	5.00	260	8.16	305	5.77
16	300	8.16	257.5	12.58	320	0.00	325	10.00
20	320	0.00	265	9.57	345	8.16	230	11.55
24	280	17.08	240	0.00	295	10.00	120	0.00

APPENDIX A.6

Peroxide Values for Refrigeration

Time (weeks)	P.V for Saran		P.V. for HB1		P.V.*** for DK11		P.V. for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	80	0.00	80	14.43	80	14.43	80	14.43
1	240	0.00	230	15.00	235	10.00	112.5	5.00
4	120	0.00	120	0.00	120	0.00	160	11.55
8	130	0.00	90	0.00	180	8.16	182.5	5.00
12	170	17.08	140	8.16	210	9.57	205	5.77
16	202	9.57	342.5	9.57	282.5	5.00	215	17.32
20	225	12.58	370	5.00	310	9.57	160	0.00
24	195	5.77	295	12.91	260	0.00	120	11.55

APPENDIX A.7

Peroxide Values for Oven Temperature

Time (weeks)	P.V for Saran		P.V.*** for HB1		P.V. for DK11		P.V. for PrnPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	80	0.00	80	0.00	80	0.00	80	2.89
1	230	17.08	215	12.91	140	5.00	215	8.16
3	270	9.57	200	5.00	150	9.57	272	8.16
4	290	0.00	160	8.16	150	8.16	287.5	5.00
6	550	9.57	210	8.16	310	5.00	327.5	9.57
8	200	0.00	240	0.00	180	20.00	340	0.00
10	220	5.00	245	5.77	200	9.57	360	8.16
12	260	8.16	270	5.00	225	5.77	345	5.77
16	245	5.77	305	5.77	320	8.16	305	12.91
20	270	17.32	335	5.77	335	0.00	250	10
24	250	11.55	300	9.57	285	17.32	220	5.77

APPENDIX A.8

Peroxide Values for Conditions in Humidity Chamber

Time (weeks)	P.V*** for Saran		P.V. for HB1		P.V. for DK11		P.V. for PrntPk	
	μ*	σ	μ	σ	μ	σ	μ	σ**
0	80	2.89	80	11.55	80	0.00	80	11.55
1	285	5.77	270	5.00	240	5.00	185	8.16
3	270	15.00	220	9.57	230	9.57	232.5	8.16
4	200	5.00	140	0.00	100	9.57	247.5	9.57
6	400	9.57	390	10.00	200	5.00	287.5	9.57
8	200	5.00	200	0.00	260	9.57	305	5.77
10	240	5.00	200	0.00	270	5.00	325	5.77
12	250	5.00	210	8.16	290	8.16	345	5.77
16	367.5	9.57	295	11.55	377.5	9.57	315	5.77
20	405	9.57	315	0.00	415.5	9.57	300	11.55
24	365	5.77	285	0.00	375	30.00	290	0.00

* μ indicates the mean of 4 replicates

** σ indicates the standard deviation of 4 replicates

***All peroxide values are in meq. of O_2/kg of peanut butter.

APPENDIX A.9

Coefficient of Variation (%) of Data Under Room Conditions

Time (weeks)	Saran		HB1		DK11		PrntPk	
	TBA	P.V.	TBA	P.V.	TBA	P.V.	TBA	P.V.
0	0.566	18.038	7.921	3.612	0.566	14.437	0.566	14.437
1	2.591	3.571	2.694	3.828	5.399	7.500	16.219	3.846
4	4.594	4.255	3.312	2.631	25.833	9.570	5.621	9.362
8	0.153	6.277	1.068	0.000	9.532	2.083	1.872	2.355
12	1.623	3.297	3.607	2.000	2.928	3.138	0.977	1.892
16	22.708	2.720	54.562	4.885	15.173	0.000	7.318	3.077
20	4.846	0.000	2.346	3.611	2.576	2.365	0.934	5.022
24	6.667	6.100	8.830	0.000	18.891	3.390	7.315	0.000
Mean C.V	5.468	5.532	10.543	2.571	10.113	5.311	5.103	4.999

APPENDIX A.10

Coefficient of Variation (%) of Data Under Refrigeration

Time (weeks)	Saran		HB1		DK11		PmtPk	
	TBA	P.V.	TBA	P.V.	TBA	P.V.	TBA	P.V.
0	7.922	0.000	7.363	18.037	7.922	18.037	7.363	18.037
1	7.615	0.000	6.442	6.522	3.300	4.255	2.552	4.444
4	3.440	0.000	2.947	0.000	2.526	0.000	17.867	7.219
8	8.068	0.000	1.494	0.000	9.488	4.533	1.087	2.740
12	3.152	10.047	1.210	5.829	1.746	4.557	1.884	2.814
16	5.971	4.738	21.400	2.794	16.891	1.770	0.943	8.056
20	4.285	5.591	6.583	1.351	6.948	3.087	3.052	0.000
24	13.909	2.959	10.876	4.376	10.618	0.000	6.017	9.625
Mean C.V	6.795	2.917	7.289	4.864	7.055	4.530	5.096	6.617

APPENDIX A.11

Coefficient of Variation (%) of Data Under Oven Temperature

Time (weeks)	Saran		HB1		DK11		PmtPk	
	TBA	P.V.	TBA	P.V.	TBA	P.V.	TBA	P.V.
0	7.363	0.000	7.363	0.000	0.566	0.000	7.922	3.612
1	3.437	7.426	1.258	6.005	2.140	3.571	8.655	3.795
3	4.470	3.544	2.822	2.500	5.736	6.380	5.680	3.000
4	0.634	0.000	1.068	5.100	1.133	5.440	8.419	1.739
6	1.127	1.740	0.474	3.886	4.812	1.613	1.392	2.922
8	4.840	0.000	1.295	0.000	1.256	11.111	0.849	0.000
10	7.796	2.273	6.003	2.355	12.608	4.785	0.441	2.267
12	1.378	3.138	3.616	1.852	24.522	2.564	4.331	1.672
16	8.875	2.355	12.318	1.892	30.231	2.550	1.794	4.233
20	2.803	6.415	0.798	1.722	3.196	0.000	2.647	4.000
24	19.073	4.620	13.681	3.190	2.451	6.077	1.274	2.623
Mean C.V	5.618	2.864	4.609	2.591	8.059	4.008	3.946	2.715

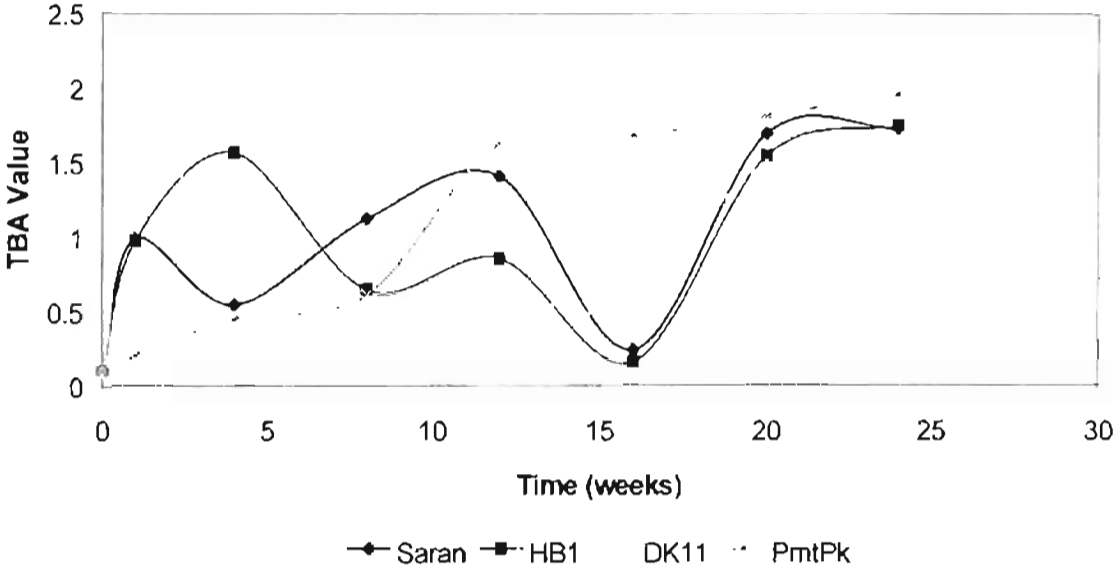
APPENDIX A.12

Coefficient of Variation (%) of Data Under Conditions in Humidity Chamber

Time (weeks)	Saran		HB1		DK11		PrntPk	
	TBA	P.V.	TBA	P.V.	TBA	P.V.	TBA	P.V.
0	0.566	3.612	7.922	14.438	7.363	0.000	7.922	14.438
1	1.037	2.024	5.394	1.852	3.270	2.083	9.717	4.411
3	1.920	5.556	5.040	4.350	4.529	4.161	6.551	3.510
4	2.713	2.500	4.745	0.000	2.016	9.570	9.869	3.867
6	1.141	2.392	1.836	2.564	6.302	2.500	0.931	3.329
8	1.548	2.500	2.730	0.000	3.473	3.681	2.861	1.892
10	4.741	2.083	0.947	0.000	2.281	1.852	0.328	1.775
12	2.508	2.000	2.156	3.886	2.274	2.814	12.329	1.672
16	11.438	2.604	16.225	3.195	12.173	2.535	12.932	1.832
20	7.273	2.363	2.892	0.000	2.844	2.303	2.983	3.850
24	7.570	1.581	5.301	0.000	5.306	8.000	1.433	0.000
Mean C.V	3.859	2.656	5.017	2.818	4.712	3.591	6.169	3.689

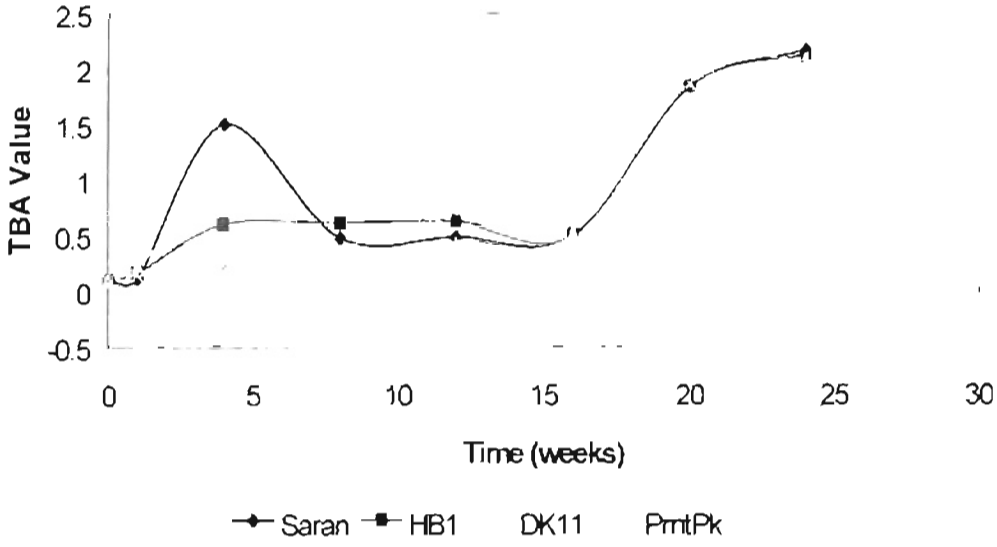
APPENDIX B.1

TBA Results Under Room Conditions



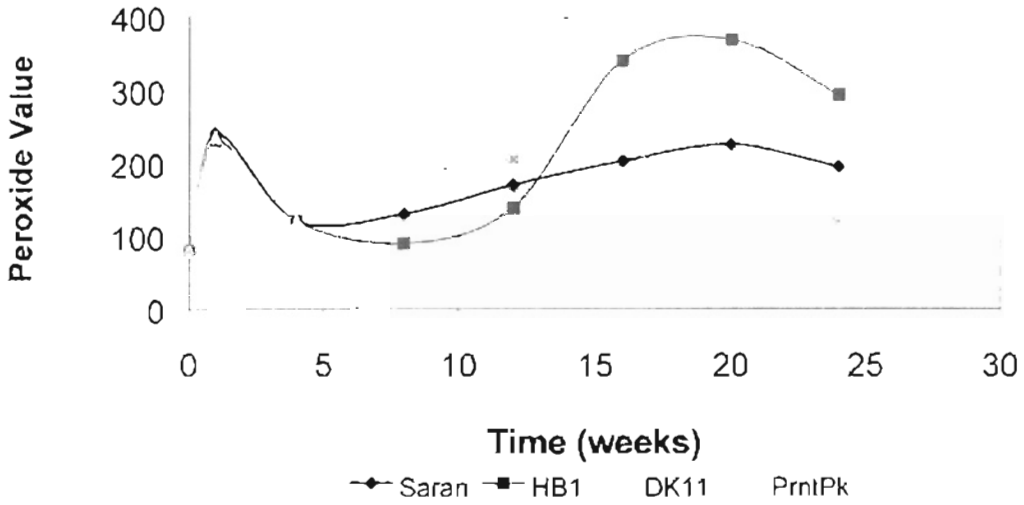
APPENDIX B.2

TBA Results Under Refrigeration



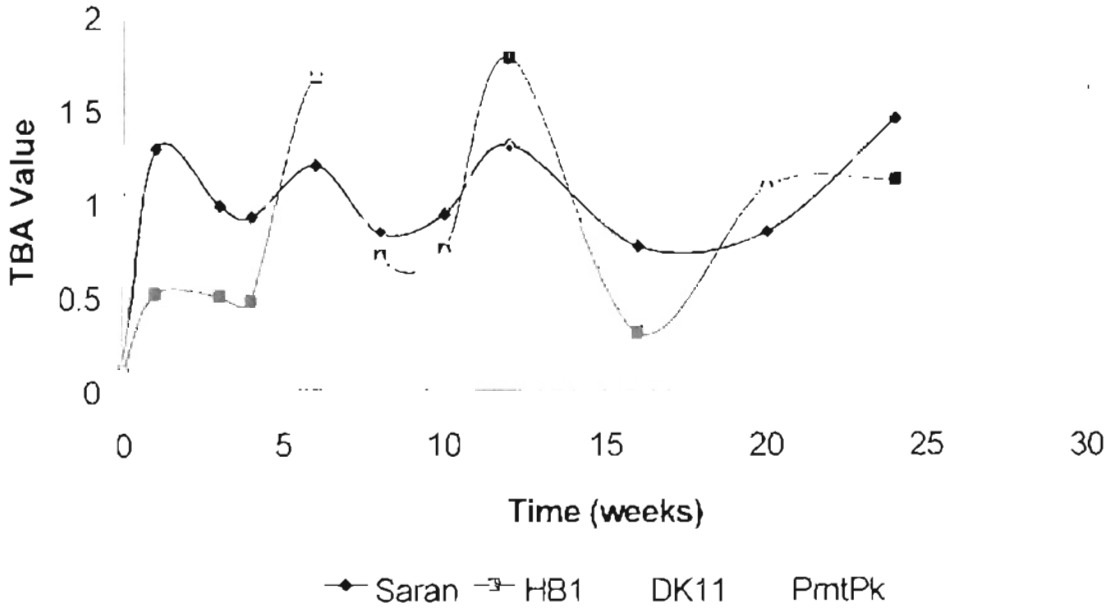
APPENDIX B.3

Peroxide Test Results Under Refrigeration



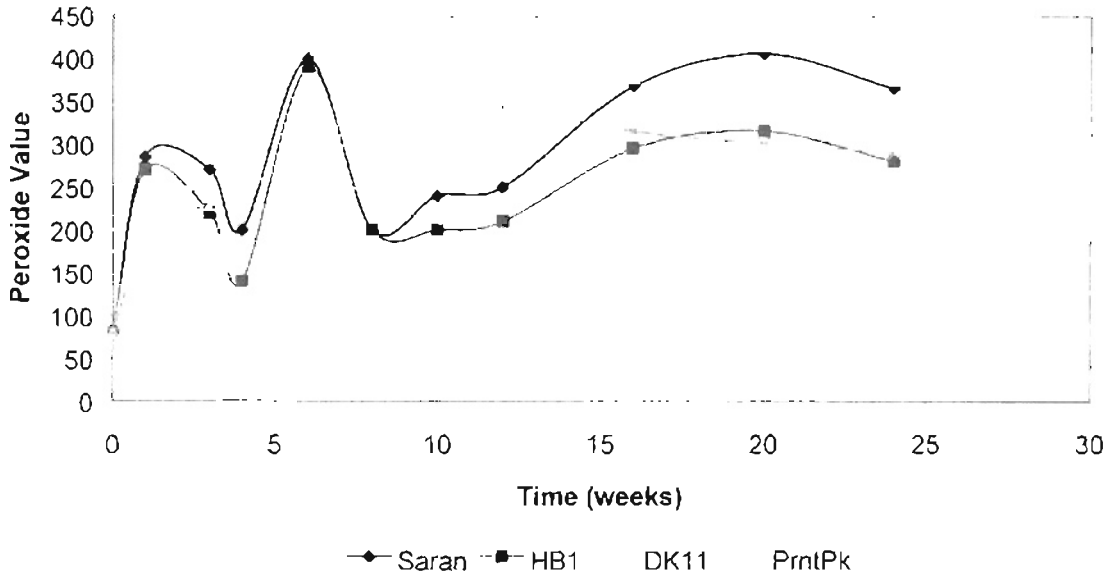
APPENDIX B.4

TBA Results Under Conditions in Humidity Chamber



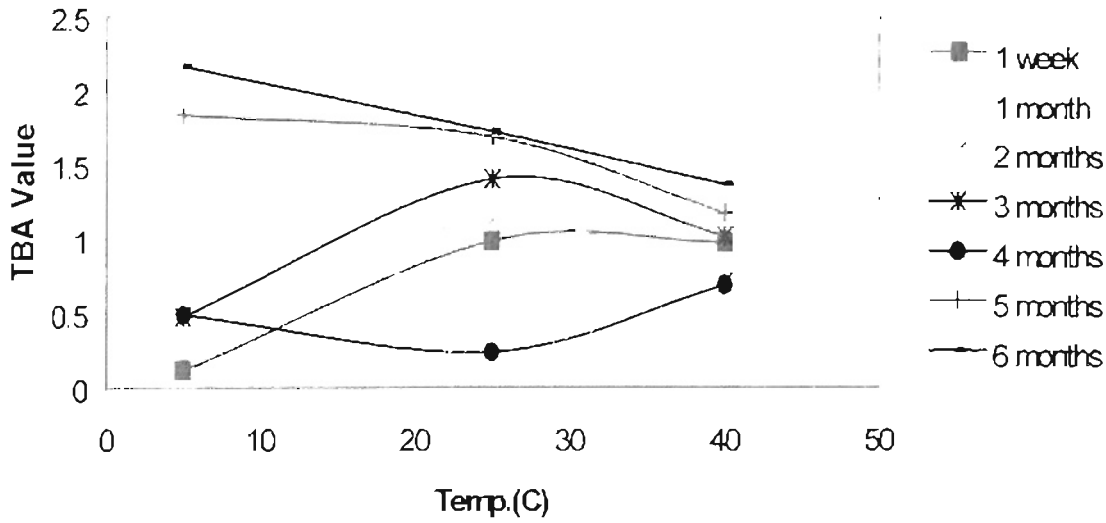
APPENDIX B.5

Peroxide Test Results Under Conditions in Humidity Chamber



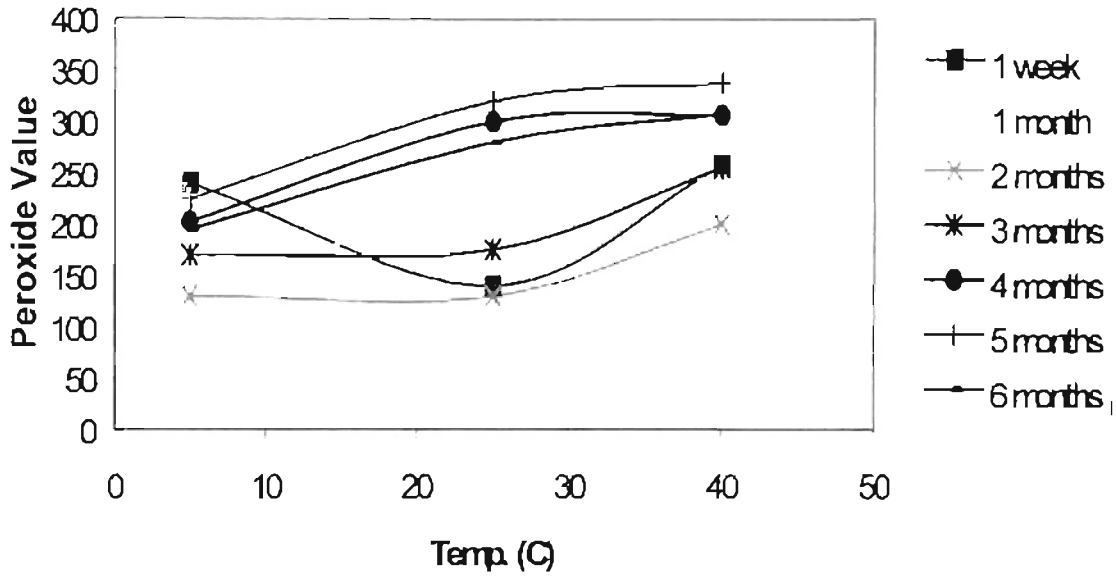
APPENDIX B.6

Temperature Effects on TBA Results for Saran



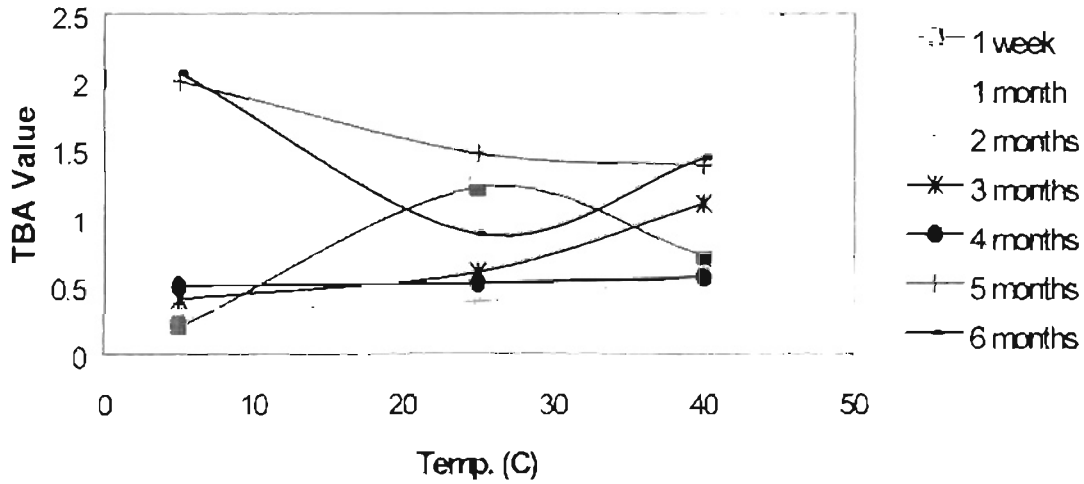
APPENDIX B.7

Temperature Effects on Peroxide Values for Saran



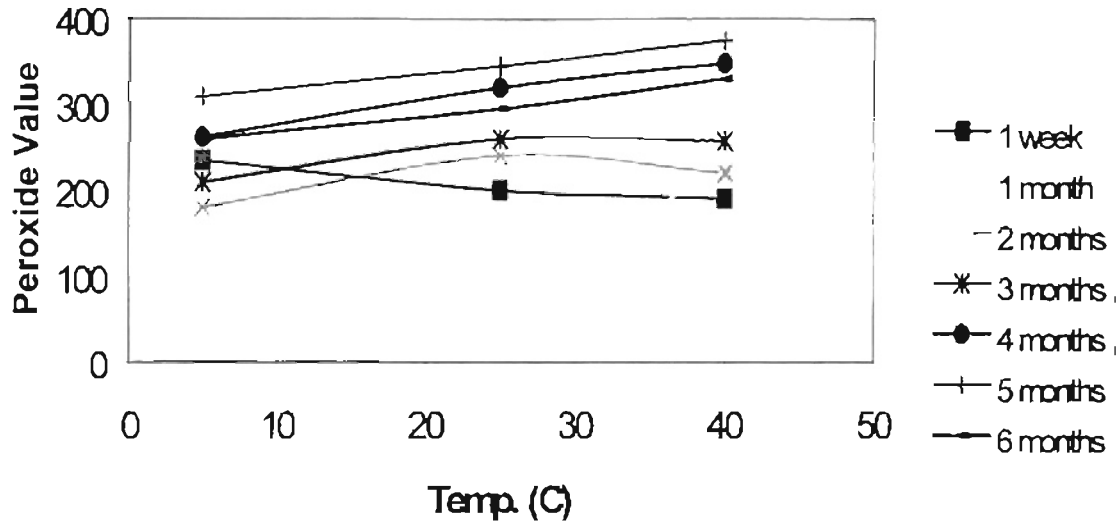
APPENDIX B.8

Temperature Effects on TBA Results for DK11 Packaging Material



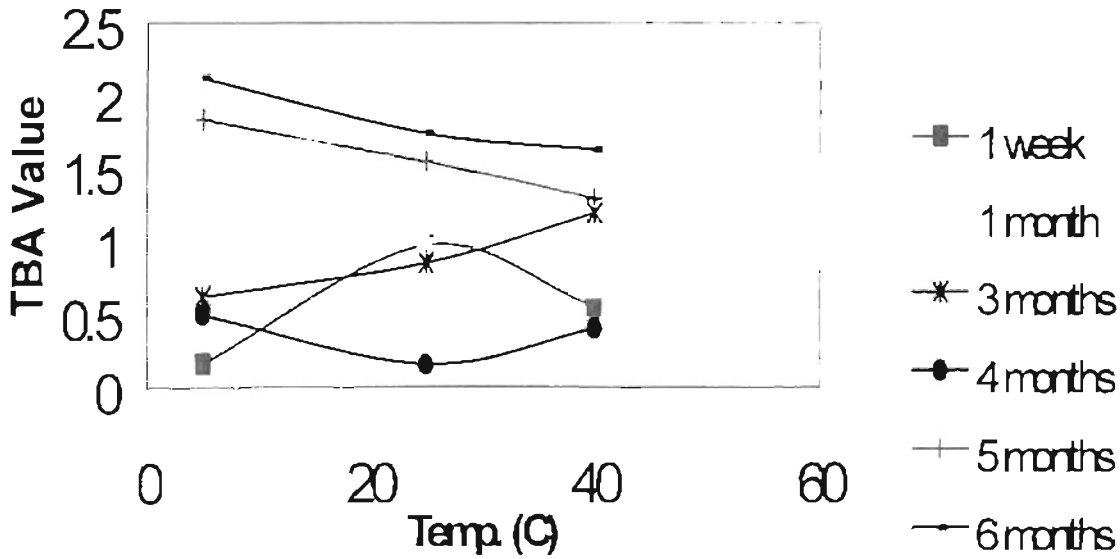
APPENDIX B.9

Temperature Effects on Peroxide Values for DK11 Packaging Material



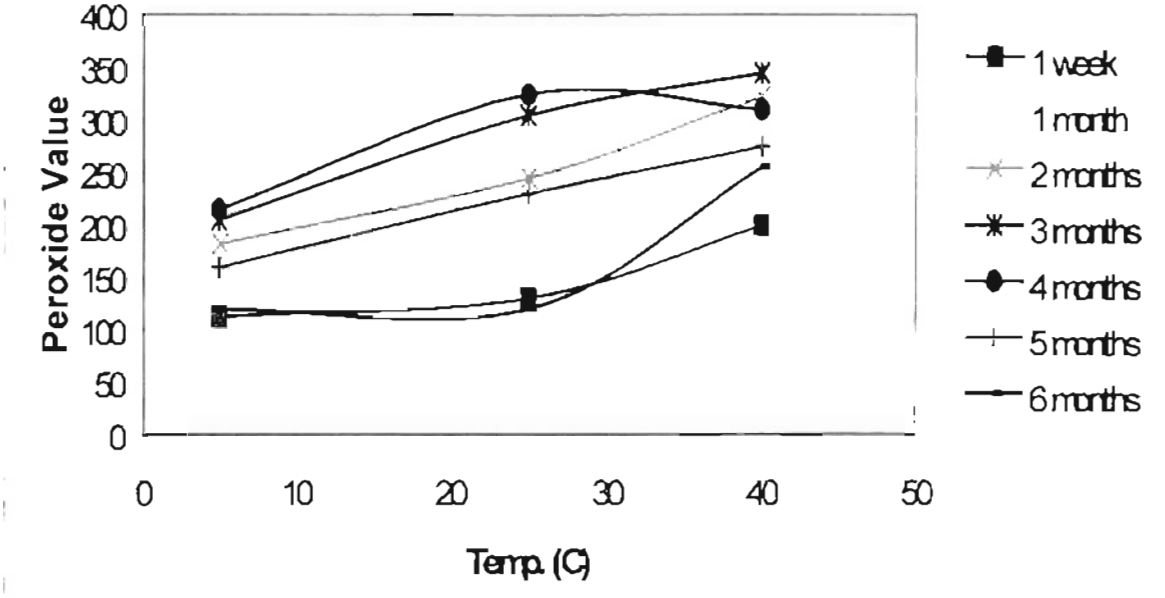
APPENDIX B.10

Temperature Effects on TBA Results for HB1 Packaging Material



APPENDIX B.11

Temperature Effects on Peroxide Values for Print Pack Material



APPENDIX C.1

Statistical Analysis of Data

SAS Code

```
dm 'log; clear; output; clear; ';

*** pbslice.sas ***;

*** This LIBNAME statement corresponds to my computer ***;
*libname mimin 'd:\temp\clients\Adhikary, Mimin\';

*** This LIBNAME statement will correspond with the A drive *;
*** Change the drive specification to correspond with the directory ***;
*** that you save these analysis files in. ***;
libname mimin 'a:\';

data mimin.pb ; set mimin.pb;
pctctrls = sqrt(pctctrl) ;
pctctrl2 = log(pctctrl + 1 ) ;
pctdiffs = sqrt(pctdiff + 1) ;
pctdiff2 = log(pctdiff + 1) ;
label pctctrl = 'Percent of Control'
    pctctrls= 'Square Root of % of Control'
    pctctrl2= 'Log of % of Control'
    pctdiff = '%Diff from Control'
    pctdiffs= 'Square Root of (%Diff from Control + 1)'
    pctdiff2= 'Log of (%Diff from Control + 1) ' ;
/*
proc gchart data = mimin.pb ;
vbar pctctrl pctdiff / group=method levels=15;
vbar pctctrl2 pctdiff2 / group=method levels=15 subgroup=storage;
vbar pctctrl2 pctdiff2 / group=method levels=15 subgroup=material;

proc univariate data=mimin.pb normal plot;
var pctctrl pctctrls pctctrl2 pctdiff pctdiffs pctdiff2;
ods listing select testsfornormality plots;
*/
proc sort data=mimin.pb; by method material storage time slice;
proc means data=mimin.pb noprint; by method material storage time slice ;
var time0 y pctctrl pctctrls pctctrl2 pctdiff pctdiffs pctdiff2;
output out=new mean=time0 y pctctrl pctctrls pctctrl2 pctdiff pctdiffs pctdiff2;

/*
proc mixed data=new covtest;
```

```
classes method material storage time slice ;
model pctctrl2 = method|material|storage|time / ddfm=kr;
random slice(method*storage) ;
repeated time  ;
lsmeans method*material*storage*time / slice= (method*material*storage
                                material*storage*time
                                method*storage*time
                                material*method*storage) ;
ods output lsmeans= plotdata ;
*/
proc sort data=new; by method;
proc mixed data=new covtest; where method='Peroxide';
TITLE 'Method = Peroxide';
classes material storage time slice ;
model pctctrl2 = material|storage|time / ddfm=kr;
random slice(material) ;
repeated time  ;
lsmeans material*storage*time / slice = (storage material time storage*time
material*time) ;

proc mixed data=new covtest; where method  'TBA';
title "Method = TBA";
classes material storage time slice ;
model pctctrl2 = material|storage|time / ddfm=kr;
random slice(material storage) ;
repeated time  ;
lsmeans material*storage*time / slice = (storage material time storage*time
material*time) ;

data plot; set mimin.plotdata;
if method = 'Peroxide' then m1=1; else m1  2;
if material='DK11' then m2=1;
 else if material='HB1' then m2=2;
 else if material='PrntPk' then m2=3;
 else if material='Saran' then m2=4;
if storage= 'HC' then s=1;
 else if storage='O' then s  2;
 else if storage='RT' then s= 3;
 else if storage='Ref' then s=4;
cat = 100*m1 + 10*m2 + s ;
cat2 = 10*m2 + s;
label cat = 'Met/Mat/S'
    cat2 = 'Mat/S';

proc format;
```

```
value catfmt 111='Per/DK11/HC' 112='Per/DK11/O' 113='Per/DK11/RT'
114='Per/DK11/Ref'
        121='Per/HB1/HC' 122='Per/HB1/O' 123 'Per/HB1/RT' 124='Per/HB1/Ref'
        131='Per/PrntPk/HC' 132='Per/PrntPk/O' 133 'Per/PrntPk/RT'
134='Per/PrntPk/Ref'
        141='Per/Saran/HC' 142='Per/Saran/O' 143='Per/Saran/RT' 144 'Per/Saran/Ref'
        211='TBA/DK11/HC' 212='TBA/DK11/O' 213='TBA/DK11/RT'
214='TBA/DK11/Ref'
        221='TBA/HB1/HC' 222='TBA/HB1/O' 223='TBA/HB1/RT'
224='TBA/HB1/Ref'
        231='TBA/PrntPk/HC' 232='TBA/PrntPk/O' 233='TBA/PrntPk/RT'
234='TBA/PrntPk/Ref'
        241='TBA/Saran/HC' 242='TBA/Saran/O' 243='TBA/Saran/RT'
244='TBA/Saran/Ref' ;
value cat2fmt 11='DK11/HC' 12='DK11/O' 13='DK11/RT' 14='DK11/Ref'
        21='HB1/HC' 22='HB1/O' 23='HB1/RT' 24='HB1/Ref'
        31='PrntPk/HC' 32='PrntPk/O' 33='PrntPk/RT' 34='PrntPk/Ref'
        41='Saran/HC' 42='Saran/O' 43='Saran/RT' 44='Saran/Ref'
        ;

proc sort data=plot; by method;
proc gplot data=plot; by method;
plot estimate*time=cat2/ vaxis=4 to 8 ;
title 'Plot of Method/Material/Storage Means Over Time';
format cat2 cat2fmt. ;
symbol1 value=circle cv=orange i=join l=1;
symbol2 value=circle cv=blue   i=join l=1;
symbol3 value=circle cv=black  i=join l  1;
symbol4 value=circle cv=red    i  join l=1;
symbol5 value=square cv=orange i=join l  1;
symbol6 value=square cv=blue   i  join l=1;
symbol7 value=square cv=black  i=join l=1;
symbol8 value=square cv=red    i=join l=1;
symbol9  value=triangle cv=orange i  join l=1;
symbol10 value=triangle cv=blue   i=join l=1;
symbol11 value=triangle cv=black  i=join l=1;
symbol12 value=triangle cv=red    i=join l=1;
symbol13 value=diamond cv=orange i=join l=1;
symbol14 value=diamond cv=blue   i=join l=1;
symbol15 value=diamond cv=black  i=join l=1;
symbol16 value=diamond cv=red    i=join l=1;


run;
quit;
```

APPENDIX C.2

Statistical Analysis of Data

SAS Output

1. Method = Peroxide 10:21 Wednesday, March 28, 2001 13

The Mixed Procedure

Model Information

Data Set	WORK.NEW
Dependent Variable	pctctrl2
Covariance Structure	Variance Components
Estimation Method	REML
Residual Variance Method	Parameter
Fixed Effects SE Method	Prasad-Rao-Jeske-Kackar-Harville
Degrees of Freedom Method	Kenward-Roger

Class Level Information

Class	Levels	Values
Material	4	DK11 HB1 PrntPk Saran
Storage	4	HC O RT Ref
time	7	1 2 3 4 5 6 7
Slice	2	1 2

Dimensions

Covariance Parameters	2
Columns in X	200
Columns in Z	8
Subjects	1
Max Obs Per Subject	224
Observations Used	224
Observations Not Used	0
Total Observations	224

Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0	1	-23.05823011	
1	1	-50.4696502	0

Convergence criteria met.

Covariance Parameter Estimates

Cov Parm	Estimate	Standard Error	Z Value	Pr Z
Slice(Material)	0.006762	0.005213	1.3	0.0973
time	0.01707	0.002322	7.35	<.0001

Fit Statistics

-2 Res Log Likelihood	-50.5
AIC (smaller is better)	-46.5
AICC (smaller is better)	-46.4
BIC (smaller is better)	-46.3

2. Method = Peroxide 10:21 Wednesday, March 28, 2001 14

The Mixed Procedure

Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Material	3	4	0.03	0.991
Storage	3	108	70.71	<.0001
Material*Storage	9	108	18.45	<.0001
time	6	108	86.37	<.0001
Material*time	18	108	20.55	<.0001
Storage*time	18	108	6.12	<.0001
Material*Storage*time	54	108	4.04	<.0001

Least Squares Means

Effect	Material	Storage	Time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	DK11	HC	1	5.5791	0.1092	35.3	51.11	<.0001
Material*Storage*time	DK11	HC	2	4.6909	0.1092	35.3	42.98	<.0001
Material*Storage*time	DK11	HC	3	5.6785	0.1092	35.3	52.02	<.0001
Material*Storage*time	DK11	HC	4	5.778	0.1092	35.3	52.94	<.0001
Material*Storage*time	DK11	HC	5	6.0411	0.1092	35.3	55.35	<.0001
Material*Storage*time	DK11	HC	6	6.0411	0.1092	35.3	55.35	<.0001
Material*Storage*time	DK11	HC	7	6.0323	0.1092	35.3	55.27	<.0001
Material*Storage*time	DK11	O	1	5.1274	0.1092	35.3	46.98	<.0001
Material*Storage*time	DK11	O	2	5.1777	0.1092	35.3	47.44	<.0001
Material*Storage*time	DK11	O	3	5.3552	0.1092	35.3	49.06	<.0001
Material*Storage*time	DK11	O	4	5.5821	0.1092	35.3	51.14	<.0001
Material*Storage*time	DK11	O	5	5.9332	0.1092	35.3	54.36	<.0001
Material*Storage*time	DK11	O	6	5.9335	0.1092	35.3	54.36	<.0001
Material*Storage*time	DK11	O	7	5.8166	0.1092	35.3	53.29	<.0001
Material*Storage*time	DK11	RT	1	5.6091	0.1092	35.3	51.39	<.0001
Material*Storage*time	DK11	RT	2	4.8806	0.1092	35.3	44.71	<.0001
Material*Storage*time	DK11	RT	3	5.7906	0.1092	35.3	53.05	<.0001
Material*Storage*time	DK11	RT	4	5.8598	0.1092	35.3	53.69	<.0001
Material*Storage*time	DK11	RT	5	6.0673	0.1092	35.3	55.59	<.0001
Material*Storage*time	DK11	RT	6	6.0671	0.1092	35.3	55.58	<.0001
Material*Storage*time	DK11	RT	7	5.9857	0.1092	35.3	54.84	<.0001
Material*Storage*time	DK11	Ref	1	5.7302	0.1092	35.3	52.5	<.0001
Material*Storage*time	DK11	Ref	2	5.062	0.1092	35.3	46.38	<.0001
Material*Storage*time	DK11	Ref	3	5.4646	0.1092	35.3	50.06	<.0001
Material*Storage*time	DK11	Ref	4	5.6299	0.1092	35.3	51.58	<.0001
Material*Storage*time	DK11	Ref	5	5.9144	0.1092	35.3	54.19	<.0001
Material*Storage*time	DK11	Ref	6	5.9141	0.1092	35.3	54.18	<.0001
Material*Storage*time	DK11	Ref	7	5.8317	0.1092	35.3	53.43	<.0001
Material*Storage*time	HB1	HC	1	5.8884	0.1092	35.3	53.95	<.0001
Material*Storage*time	HB1	HC	2	5.2436	0.1092	35.3	48.04	<.0001
Material*Storage*time	HB1	HC	3	5.5987	0.1092	35.3	51.29	<.0001
Material*Storage*time	HB1	HC	4	5.6468	0.1092	35.3	51.73	<.0001
Material*Storage*time	HB1	HC	5	6.5126	0.1092	35.3	59.67	<.0001
Material*Storage*time	HB1	HC	6	6.5128	0.1092	35.3	59.67	<.0001
Material*Storage*time	HB1	HC	7	5.9341	0.1092	35.3	54.37	<.0001
Material*Storage*time	HB1	O	1	5.4788	0.1092	35.3	50.2	<.0001
Material*Storage*time	HB1	O	2	5.1852	0.1092	35.3	47.5	<.0001
Material*Storage*time	HB1	O	3	5.5897	0.1092	35.3	51.21	<.0001

Material*Storage*time	HB1	O	4	5.7162	0.1092	35.3	52.37	<.0001
Material*Storage*time	HB1	O	5	5.8285	0.1092	35.3	53.4	<.0001
Material*Storage*time	HB1	O	6	5.8285	0.1092	35.3	53.4	<.0001
Material*Storage*time	HB1	O	7	5.8034	0.1092	35.3	53.17	<.0001
Material*Storage*time	HB1	RT	1	5.6483	0.1092	35.3	51.75	<.0001
Material*Storage*time	HB1	RT	2	5.3983	0.1092	35.3	49.46	<.0001

3. Method = Peroxide 10:21 Wednesday, March 28, 2001 15

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	HB1	RT	3	5.6182	0.1092	35.3	51.47	<.0001
Material*Storage*time	HB1	RT	4	5.6487	0.1092	35.3	51.75	<.0001
Material*Storage*time	HB1	RT	5	5.6874	0.1092	35.3	52.11	<.0001
Material*Storage*time	HB1	RT	6	5.6878	0.1092	35.3	52.11	<.0001
Material*Storage*time	HB1	RT	7	5.5315	0.1092	35.3	50.68	<.0001
Material*Storage*time	HB1	Ref	1	5.7187	0.1092	35.3	52.39	<.0001
Material*Storage*time	HB1	Ref	2	5.062	0.1092	35.3	46.38	<.0001
Material*Storage*time	HB1	Ref	3	4.7765	0.1092	35.3	43.76	<.0001
Material*Storage*time	HB1	Ref	4	5.2139	0.1092	35.3	47.77	<.0001
Material*Storage*time	HB1	Ref	5	6.1063	0.1092	35.3	55.94	<.0001
Material*Storage*time	HB1	Ref	6	6.1065	0.1092	35.3	55.95	<.0001
Material*Storage*time	HB1	Ref	7	5.9569	0.1092	35.3	54.58	<.0001
Material*Storage*time	PrntPk	HC	1	5.6932	0.1092	35.3	52.16	<.0001
Material*Storage*time	PrntPk	HC	2	5.8105	0.1092	35.3	53.23	<.0001
Material*Storage*time	PrntPk	HC	3	6.0193	0.1092	35.3	55.15	<.0001
Material*Storage*time	PrntPk	HC	4	6.1423	0.1092	35.3	56.27	<.0001
Material*Storage*time	PrntPk	HC	5	6.0515	0.1092	35.3	55.44	<.0001
Material*Storage*time	PrntPk	HC	6	6.0024	0.1092	35.3	54.99	<.0001
Material*Storage*time	PrntPk	HC	7	5.9691	0.1092	35.3	54.69	<.0001
Material*Storage*time	PrntPk	O	1	5.6585	0.1092	35.3	51.84	<.0001
Material*Storage*time	PrntPk	O	2	5.7981	0.1092	35.3	53.12	<.0001
Material*Storage*time	PrntPk	O	3	5.9655	0.1092	35.3	54.65	<.0001
Material*Storage*time	PrntPk	O	4	5.9799	0.1092	35.3	54.79	<.0001
Material*Storage*time	PrntPk	O	5	5.8564	0.1092	35.3	53.65	<.0001
Material*Storage*time	PrntPk	O	6	5.6382	0.1092	35.3	51.65	<.0001
Material*Storage*time	PrntPk	O	7	5.5084	0.1092	35.3	50.47	<.0001

Material*Storage*time	PrntPk	RT	1	5.2608	0.1092	35.3	48.2	<.0001
Material*Storage*time	PrntPk	RT	2	5.5176	0.1092	35.3	50.55	<.0001
Material*Storage*time	PrntPk	RT	3	5.8008	0.1092	35.3	53.14	<.0001
Material*Storage*time	PrntPk	RT	4	6.0193	0.1092	35.3	55.15	<.0001
Material*Storage*time	PrntPk	RT	5	6.0824	0.1092	35.3	55.72	<.0001
Material*Storage*time	PrntPk	RT	6	5.737	0.1092	35.3	52.56	<.0001
Material*Storage*time	PrntPk	RT	7	5.0904	0.1092	35.3	46.64	<.0001
Material*Storage*time	PrntPk	Ref	1	5.0819	0.1092	35.3	46.56	<.0001
Material*Storage*time	PrntPk	Ref	2	5.3462	0.1092	35.3	48.98	<.0001
Material*Storage*time	PrntPk	Ref	3	5.4788	0.1092	35.3	50.19	<.0001
Material*Storage*time	PrntPk	Ref	4	5.5946	0.1092	35.3	51.26	<.0001
Material*Storage*time	PrntPk	Ref	5	5.6398	0.1092	35.3	51.67	<.0001
Material*Storage*time	PrntPk	Ref	6	5.4653	0.1092	35.3	50.07	<.0001
Material*Storage*time	PrntPk	Ref	7	5.0586	0.1092	35.3	46.34	<.0001
Material*Storage*time	Saran	HC	1	5.7893	0.1092	35.3	53.04	<.0001
Material*Storage*time	Saran	HC	2	5.4239	0.1092	35.3	49.69	<.0001
Material*Storage*time	Saran	HC	3	5.4239	0.1092	35.3	49.69	<.0001
Material*Storage*time	Saran	HC	4	5.6687	0.1092	35.3	51.93	<.0001
Material*Storage*time	Saran	HC	5	6.0428	0.1092	35.3	55.36	<.0001
Material*Storage*time	Saran	HC	6	6.0428	0.1092	35.3	55.36	<.0001
Material*Storage*time	Saran	HC	7	6.0361	0.1092	35.3	55.3	<.0001
Material*Storage*time	Saran	O	1	5.8804	0.1092	35.3	53.87	<.0001
Material*Storage*time	Saran	O	2	6.1029	0.1092	35.3	55.91	<.0001
Material*Storage*time	Saran	O	3	5.7323	0.1092	35.3	52.52	<.0001
Material*Storage*time	Saran	O	4	5.9936	0.1092	35.3	54.91	<.0001
Material*Storage*time	Saran	O	5	5.9345	0.1092	35.3	54.37	<.0001
Material*Storage*time	Saran	O	6	5.9328	0.1092	35.3	54.35	<.0001
Material*Storage*time	Saran	O	7	5.954	0.1092	35.3	54.55	<.0001
Material*Storage*time	Saran	RT	1	5.2324	0.1092	35.3	47.94	<.0001
Material*Storage*time	Saran	RT	2	5.7303	0.1092	35.3	52.5	<.0001
Material*Storage*time	Saran	RT	3	5.1401	0.1092	35.3	47.09	<.0001
Material*Storage*time	Saran	RT	4	5.4369	0.1092	35.3	49.81	<.0001

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	Saran	RT	5	5.9742	0.1092	35.3	54.73	<.0001
Material*Storage*time	Saran	RT	6	5.9744	0.1092	35.3	54.74	<.0001
Material*Storage*time	Saran	RT	7	5.8953	0.1092	35.3	54.01	<.0001
Material*Storage*time	Saran	Ref	1	5.6467	0.1092	35.3	51.73	<.0001
Material*Storage*time	Saran	Ref	2	4.9571	0.1092	35.3	45.41	<.0001
Material*Storage*time	Saran	Ref	3	5.0366	0.1092	35.3	46.14	<.0001
Material*Storage*time	Saran	Ref	4	5.3141	0.1092	35.3	48.69	<.0001
Material*Storage*time	Saran	Ref	5	5.4766	0.1092	35.3	50.17	<.0001
Material*Storage*time	Saran	Ref	6	5.476	0.1092	35.3	50.17	<.0001
Material*Storage*time	Saran	Ref	7	5.4395	0.1092	35.3	49.84	<.0001

Tests of Effect Slices

Effect	Material	Storage	time	Num DF	Den DF	F Value	Pr > F
Material*Storage*time		HC		27	77.4	14.4	<.0001
Material*Storage*time		O		27	77.4	6.22	<.0001
Material*Storage*time		RT		27	77.4	11.5	<.0001
Material*Storage*time		Ref		27	77.4	13.3	<.0001
Material*Storage*time	DK11			27	108	16.68	<.0001
Material*Storage*time	HB1			27	108	17.41	<.0001
Material*Storage*time	PrntPk			27	108	11.78	<.0001
Material*Storage*time	Saran			27	108	13.19	<.0001
Material*Storage*time			1	15	65.4	6.66	<.0001
Material*Storage*time			2	15	65.4	11.35	<.0001
Material*Storage*time			3	15	65.4	9.98	<.0001
Material*Storage*time			4	15	65.4	5.88	<.0001
Material*Storage*time			5	15	65.4	5.82	<.0001
Material*Storage*time			6	15	65.4	6.54	<.0001
Material*Storage*time			7	15	65.4	8.73	<.0001
Material*Storage*time		HC	1	3	35.3	1.47	0.2396
Material*Storage*time		HC	2	3	35.3	18.18	<.0001
Material*Storage*time		HC	3	3	35.3	5.24	0.0043

Material*Storage*time		HC	4	3	35.3	4.42	0.0097
Material*Storage*time		HC	5	3	35.3	4.59	0.0082
Material*Storage*time		HC	6	3	35.3	4.94	0.0057
Material*Storage*time		HC	7	3	35.3	0.21	0.89
Material*Storage*time		O	1	3	35.3	8.5	0.0002
Material*Storage*time		O	2	3	35.3	17.85	<.0001
Material*Storage*time		O	3	3	35.3	5.49	0.0033
Material*Storage*time		O	4	3	35.3	3.44	0.027
Material*Storage*time		O	5	3	35.3	0.24	0.8645
Material*Storage*time		O	6	3	35.3	1.62	0.2012
Material*Storage*time		O	7	3	35.3	2.95	0.0457
Material*Storage*time		RT	1	3	35.3	4.12	0.0132
Material*Storage*time		RT	2	3	35.3	10.95	<.0001
Material*Storage*time		RT	3	3	35.3	8.05	0.0003
Material*Storage*time		RT	4	3	35.3	5.39	0.0037
Material*Storage*time		RT	5	3	35.3	2.82	0.0529
Material*Storage*time		RT	6	3	35.3	2.81	0.0533
Material*Storage*time		RT	7	3	35.3	13.93	<.0001
Material*Storage*time		Ref	1	3	35.3	8.09	0.0003
Material*Storage*time		Ref	2	3	35.3	2.34	0.0897
Material*Storage*time		Ref	3	3	35.3	9.89	<.0001
Material*Storage*time		Ref	4	3	35.3	3.55	0.024
Material*Storage*time		Ref	5	3	35.3	6.61	0.0012

5. Method = Peroxide 10:21 Wednesday, March 28, 2001 17

The Mixed Procedure

Tests of Effect Slices

Effect	Material	Storage	time	Num DF	Den DF	F Value	Pr > F
Material*Storage*time		Ref	6	3	35.3	8.67	0.0002
Material*Storage*time		Ref	7	3	35.3	13.9	<.0001
Material*Storage*time	DK11		1	3	108	8.18	<.0001
Material*Storage*time	DK11		2	3	108	5.32	0.0018
Material*Storage*time	DK11		3	3	108	4.6	0.0046
Material*Storage*time	DK11		4	3	108	1.95	0.1265
Material*Storage*time	DK11		5	3	108	0.68	0.5634
Material*Storage*time	DK11		6	3	108	0.68	0.5639
Material*Storage*time	DK11		7	3	108	1.38	0.2522
Material*Storage*time	HB1		1	3	108	3.37	0.0211

Material*Storage*time	HB1		2	3	108	2.29	0.0829
Material*Storage*time	HB1		3	3	108	19.99	<.0001
Material*Storage*time	HB1		4	3	108	6.23	0.0006
Material*Storage*time	HB1		5	3	108	15.49	<.0001
Material*Storage*time	HB1		6	3	108	15.49	<.0001
Material*Storage*time	HB1		7	3	108	4.47	0.0053
Material*Storage*time	PrntPk		1	3	108	10.59	<.0001
Material*Storage*time	PrntPk		2	3	108	6	0.0008
Material*Storage*time	PrntPk		3	3	108	6.94	0.0003
Material*Storage*time	PrntPk		4	3	108	6.56	0.0004
Material*Storage*time	PrntPk		5	3	108	4.91	0.0031
Material*Storage*time	PrntPk		6	3	108	5.91	0.0009
Material*Storage*time	PrntPk		7	3	108	21.4	<.0001
Material*Storage*time	Saran		1	3	108	9.62	<.0001
Material*Storage*time	Saran		2	3	108	27.56	<.0001
Material*Storage*time	Saran		3	3	108	11.44	<.0001
Material*Storage*time	Saran		4	3	108	10.47	<.0001
Material*Storage*time	Saran		5	3	108	7.77	<.0001
Material*Storage*time	Saran		6	3	108	7.78	<.0001
Material*Storage*time	Saran		7	3	108	8.38	<.0001

The Mixed Procedure

Model Information

Data Set	WORK.NEW
Dependent Variable	petetrl2
Covariance Structure	Variance Components
Estimation Method	REML
Residual Variance Method	Parameter
Fixed Effects SE Method	Prasad-Rao-Jeske-Kackar-Harville
Degrees of Freedom Method	Kenward-Roger

Class Level Information

Class	Levels	Values
Material	4	DK11 HB1 PrntPk Saran
Storage	4	HC O RT Ref
time	7	1 2 3 4 5 6 7
Slice	2	1 2

Dimensions

Covariance Parameters	2
Columns in X	200
Columns in Z	32
Subjects	1
Max Obs Per Subject	224
Observations Used	224
Observations Not Used	0
Total Observations	224

Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0	1	-125.1095158	
1	1	-155.4975012	0

Convergence criteria met.

Covariance Parameter Estimates

Cov Parm	Estimate	Standard Error	Z Value	Pr Z
Slice(Material*Storage)	0.003899	0.00167	2.34	0.0098
time	0.005681	0.00082	6.93	<.0001

Fit Statistics

-2 Res Log Likelihood	-155.5
AIC (smaller is better)	-151.5
AICC (smaller is better)	-151.4
BIC (smaller is better)	-148.6

The Mixed Procedure

Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Material	3	16	10.51	0.0005
Storage	3	16	40.68	<.0001
Material*Storage	9	16	6.33	0.0007
time	6	96	1400.49	<.0001
Material*time	18	96	195.96	<.0001
Storage*time	18	96	195.35	<.0001
Material*Storage*time	54	96	50.48	<.0001

Least Squares Means

Effect	Material	Storage	time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	DK11	HC	1	6.8992	0.06921	56.2	99.69	<.0001
Material*Storage*time	DK11	HC	2	6.5712	0.06921	56.2	94.95	<.0001
Material*Storage*time	DK11	HC	3	6.5007	0.06921	56.2	93.93	<.0001
Material*Storage*time	DK11	HC	4	7.3277	0.06921	56.2	105.88	<.0001
Material*Storage*time	DK11	HC	5	6.8556	0.06921	56.2	99.06	<.0001
Material*Storage*time	DK11	HC	6	6.9523	0.06921	56.2	100.45	<.0001
Material*Storage*time	DK11	HC	7	7.1339	0.06921	56.2	103.08	<.0001
Material*Storage*time	DK11	O	1	6.0382	0.06921	56.2	87.24	<.0001
Material*Storage*time	DK11	O	2	6.8285	0.06921	56.2	98.66	<.0001
Material*Storage*time	DK11	O	3	6.1864	0.06921	56.2	89.39	<.0001
Material*Storage*time	DK11	O	4	6.45	0.06921	56.2	93.2	<.0001
Material*Storage*time	DK11	O	5	4.8696	0.06921	56.2	70.36	<.0001
Material*Storage*time	DK11	O	6	7.4616	0.06921	56.2	107.81	<.0001

Material*Storage*time	DK11	O	7	7.3903	0.06921	56.2	106.78	<.0001
Material*Storage*time	DK11	RT	1	7.132	0.06921	56.2	103.05	<.0001
Material*Storage*time	DK11	RT	2	7.19	0.06921	56.2	103.89	<.0001
Material*Storage*time	DK11	RT	3	5.9776	0.06921	56.2	86.37	<.0001
Material*Storage*time	DK11	RT	4	6.4252	0.06921	56.2	92.84	<.0001
Material*Storage*time	DK11	RT	5	6.273	0.06921	56.2	90.64	<.0001
Material*Storage*time	DK11	RT	6	7.3222	0.06921	56.2	105.8	<.0001
Material*Storage*time	DK11	RT	7	6.7955	0.06921	56.2	98.19	<.0001
Material*Storage*time	DK11	Ref	1	5.2822	0.06921	56.2	76.32	<.0001
Material*Storage*time	DK11	Ref	2	6.8489	0.06921	56.2	98.96	<.0001
Material*Storage*time	DK11	Ref	3	5.7666	0.06921	56.2	83.32	<.0001
Material*Storage*time	DK11	Ref	4	5.9786	0.06921	56.2	86.38	<.0001
Material*Storage*time	DK11	Ref	5	6.1788	0.06921	56.2	89.28	<.0001
Material*Storage*time	DK11	Ref	6	7.5669	0.06921	56.2	109.33	<.0001
Material*Storage*time	DK11	Ref	7	7.5957	0.06921	56.2	109.75	<.0001
Material*Storage*time	HB1	HC	1	6.1959	0.06921	56.2	89.52	<.0001
Material*Storage*time	HB1	HC	2	6.1177	0.06921	56.2	88.39	<.0001
Material*Storage*time	HB1	HC	3	6.5246	0.06921	56.2	94.27	<.0001
Material*Storage*time	HB1	HC	4	7.4404	0.06921	56.2	107.5	<.0001
Material*Storage*time	HB1	HC	5	5.6671	0.06921	56.2	81.88	<.0001
Material*Storage*time	HB1	HC	6	6.941	0.06921	56.2	100.29	<.0001
Material*Storage*time	HB1	HC	7	6.9777	0.06921	56.2	100.82	<.0001
Material*Storage*time	HB1	O	1	6.3574	0.06921	56.2	91.86	<.0001
Material*Storage*time	HB1	O	2	6.7589	0.06921	56.2	97.66	<.0001
Material*Storage*time	HB1	O	3	6.2624	0.06921	56.2	90.48	<.0001
Material*Storage*time	HB1	O	4	6.3973	0.06921	56.2	92.43	<.0001
Material*Storage*time	HB1	O	5	6.1869	0.06921	56.2	89.39	<.0001
Material*Storage*time	HB1	O	6	7.2824	0.06921	56.2	105.22	<.0001
Material*Storage*time	HB1	O	7	7.6238	0.06921	56.2	110.15	<.0001
Material*Storage*time	HB1	RT	1	6.8473	0.06921	56.2	98.94	<.0001
Material*Storage*time	HB1	RT	2	7.3181	0.06921	56.2	105.74	<.0001

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	HB1	RT	3	6.4284	0.06921	56.2	92.88	<.0001
Material*Storage*time	HB1	RT	4	6.7134	0.06921	56.2	97	<.0001
Material*Storage*time	HB1	RT	5	4.9194	0.06921	56.2	71.08	<.0001
Material*Storage*time	HB1	RT	6	7.305	0.06921	56.2	105.55	<.0001
Material*Storage*time	HB1	RT	7	7.4245	0.06921	56.2	107.27	<.0001
Material*Storage*time	HB1	Ref	1	5.055	0.06921	56.2	73.04	<.0001
Material*Storage*time	HB1	Ref	2	6.3625	0.06921	56.2	91.93	<.0001
Material*Storage*time	HB1	Ref	3	6.3718	0.06921	56.2	92.06	<.0001
Material*Storage*time	HB1	Ref	4	6.3925	0.06921	56.2	92.36	<.0001
Material*Storage*time	HB1	Ref	5	6.1578	0.06921	56.2	88.97	<.0001
Material*Storage*time	HB1	Ref	6	7.4733	0.06921	56.2	107.98	<.0001
Material*Storage*time	HB1	Ref	7	7.6151	0.06921	56.2	110.03	<.0001
Material*Storage*time	PrntPk	HC	1	6.2561	0.06921	56.2	90.39	<.0001
Material*Storage*time	PrntPk	HC	2	6.3775	0.06921	56.2	92.15	<.0001
Material*Storage*time	PrntPk	HC	3	6.6449	0.06921	56.2	96.01	<.0001
Material*Storage*time	PrntPk	HC	4	7.3714	0.06921	56.2	106.51	<.0001
Material*Storage*time	PrntPk	HC	5	6.9867	0.06921	56.2	100.95	<.0001
Material*Storage*time	PrntPk	HC	6	7.1868	0.06921	56.2	103.84	<.0001
Material*Storage*time	PrntPk	HC	7	7.416	0.06921	56.2	107.15	<.0001
Material*Storage*time	PrntPk	O	1	5.8336	0.06921	56.2	84.29	<.0001
Material*Storage*time	PrntPk	O	2	6.11	0.06921	56.2	88.28	<.0001
Material*Storage*time	PrntPk	O	3	6.522	0.06921	56.2	94.23	<.0001
Material*Storage*time	PrntPk	O	4	7.2489	0.06921	56.2	104.74	<.0001
Material*Storage*time	PrntPk	O	5	7.4624	0.06921	56.2	107.82	<.0001
Material*Storage*time	PrntPk	O	6	7.4765	0.06921	56.2	108.03	<.0001
Material*Storage*time	PrntPk	O	7	7.5131	0.06921	56.2	108.56	<.0001
Material*Storage*time	PrntPk	RT	1	5.453	0.06921	56.2	78.79	<.0001
Material*Storage*time	PrntPk	RT	2	6.0977	0.06921	56.2	88.1	<.0001
Material*Storage*time	PrntPk	RT	3	6.4386	0.06921	56.2	93.03	<.0001
Material*Storage*time	PrntPk	RT	4	7.421	0.06921	56.2	107.22	<.0001
Material*Storage*time	PrntPk	RT	5	7.4492	0.06921	56.2	107.63	<.0001
Material*Storage*time	PrntPk	RT	6	7.5269	0.06921	56.2	108.75	<.0001
Material*Storage*time	PrntPk	RT	7	7.6031	0.06921	56.2	109.86	<.0001
Material*Storage*time	PrntPk	Ref	1	4.9819	0.06921	56.2	71.98	<.0001

Material*Storage*time	PrntPk	Ref	2	5.3046	0.06921	56.2	76.64	<.0001
Material*Storage*time	PrntPk	Ref	3	5.9267	0.06921	56.2	85.63	<.0001
Material*Storage*time	PrntPk	Ref	4	6.5413	0.06921	56.2	94.51	<.0001
Material*Storage*time	PrntPk	Ref	5	7.4733	0.06921	56.2	107.98	<.0001
Material*Storage*time	PrntPk	Ref	6	7.4854	0.06921	56.2	108.15	<.0001
Material*Storage*time	PrntPk	Ref	7	7.5635	0.06921	56.2	109.28	<.0001
Material*Storage*time	Saran	HC	1	7.1822	0.06921	56.2	103.77	<.0001
Material*Storage*time	Saran	HC	2	6.8437	0.06921	56.2	98.88	<.0001
Material*Storage*time	Saran	HC	3	6.7445	0.06921	56.2	97.45	<.0001
Material*Storage*time	Saran	HC	4	7.1925	0.06921	56.2	103.92	<.0001
Material*Storage*time	Saran	HC	5	6.653	0.06921	56.2	96.13	<.0001
Material*Storage*time	Saran	HC	6	6.7532	0.06921	56.2	97.58	<.0001
Material*Storage*time	Saran	HC	7	7.2963	0.06921	56.2	105.42	<.0001
Material*Storage*time	Saran	O	1	6.4734	0.06921	56.2	93.53	<.0001
Material*Storage*time	Saran	O	2	6.3945	0.06921	56.2	92.39	<.0001
Material*Storage*time	Saran	O	3	6.3436	0.06921	56.2	91.66	<.0001
Material*Storage*time	Saran	O	4	6.5615	0.06921	56.2	94.81	<.0001
Material*Storage*time	Saran	O	5	6.3901	0.06921	56.2	92.33	<.0001
Material*Storage*time	Saran	O	6	7.2775	0.06921	56.2	105.15	<.0001
Material*Storage*time	Saran	O	7	7.1118	0.06921	56.2	102.76	<.0001
Material*Storage*time	Saran	RT	1	6.9218	0.06921	56.2	100.01	<.0001
Material*Storage*time	Saran	RT	2	6.3221	0.06921	56.2	91.35	<.0001
Material*Storage*time	Saran	RT	3	7.0466	0.06921	56.2	101.82	<.0001
Material*Storage*time	Saran	RT	4	7.2737	0.06921	56.2	105.1	<.0001

9. Method = TBA 10:21 Wednesday, March 28, 2001 21

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	time	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage*time	Saran	RT	5	5.4866	0.06921	56.2	79.28	<.0001
Material*Storage*time	Saran	RT	6	7.4579	0.06921	56.2	107.76	<.0001
Material*Storage*time	Saran	RT	7	7.4772	0.06921	56.2	108.04	<.0001
Material*Storage*time	Saran	Ref	1	4.6861	0.06921	56.2	67.71	<.0001
Material*Storage*time	Saran	Ref	2	7.2757	0.06921	56.2	105.12	<.0001
Material*Storage*time	Saran	Ref	3	6.1166	0.06921	56.2	88.38	<.0001
Material*Storage*time	Saran	Ref	4	6.1355	0.06921	56.2	88.65	<.0001
Material*Storage*time	Saran	Ref	5	6.1557	0.06921	56.2	88.94	<.0001
Material*Storage*time	Saran	Ref	6	7.4761	0.06921	56.2	108.02	<.0001
Material*Storage*time	Saran	Ref	7	7.6341	0.06921	56.2	110.3	<.0001

Tests of Effect Slices

Effect	Material	Storage	time	Num DF	Den DF	F Value	Pr > F
Material*Storage*time		HC		27	92.6	59.24	<.0001
Material*Storage*time		O		27	92.6	137.65	<.0001
Material*Storage*time		RT		27	92.6	176.38	<.0001
Material*Storage*time		Ref		27	92.6	295.6	<.0001
Material*Storage*time	DK11			27	92.6	153.46	<.0001
Material*Storage*time	HB1			27	92.6	168.97	<.0001
Material*Storage*time	PrntPk			27	92.6	206.74	<.0001
Material*Storage*time	Saran			27	92.6	143	<.0001
Material*Storage*time			1	15	56.2	137.11	<.0001
Material*Storage*time			2	15	56.2	57.07	<.0001
Material*Storage*time			3	15	56.2	21.85	<.0001
Material*Storage*time			4	15	56.2	53.15	<.0001
Material*Storage*time			5	15	56.2	139.69	<.0001
Material*Storage*time			6	15	56.2	12	<.0001
Material*Storage*time			7	15	56.2	13.67	<.0001
Material*Storage*time		HC	1	3	56.2	49.1	<.0001
Material*Storage*time		HC	2	3	56.2	19.65	<.0001
Material*Storage*time		HC	3	3	56.2	2.67	0.0561

Material*Storage*time		HC	4	3	56.2	2.28	0.0892
Material*Storage*time		HC	5	3	56.2	74.74	<.0001
Material*Storage*time		HC	6	3	56.2	6.58	0.0007
Material*Storage*time		HC	7	3	56.2	7.62	0.0002
Material*Storage*time		O	1	3	56.2	17.92	<.0001
Material*Storage*time		O	2	3	56.2	23.39	<.0001
Material*Storage*time		O	3	3	56.2	4.33	0.0082
Material*Storage*time		O	4	3	56.2	32.68	<.0001
Material*Storage*time		O	5	3	56.2	236.38	<.0001
Material*Storage*time		O	6	3	56.2	2.5	0.069
Material*Storage*time		O	7	3	56.2	10.14	<.0001
Material*Storage*time		RT	1	3	56.2	122.68	<.0001
Material*Storage*time		RT	2	3	56.2	78.2	<.0001
Material*Storage*time		RT	3	3	56.2	40.19	<.0001
Material*Storage*time		RT	4	3	56.2	45.77	<.0001
Material*Storage*time		RT	5	3	56.2	250.66	<.0001
Material*Storage*time		RT	6	3	56.2	2.4	0.0772
Material*Storage*time		RT	7	3	56.2	27.19	<.0001
Material*Storage*time		Ref	1	3	56.2	12.63	<.0001
Material*Storage*time		Ref	2	3	56.2	150.34	<.0001
Material*Storage*time		Ref	3	3	56.2	14.16	<.0001
Material*Storage*time		Ref	4	3	56.2	13.32	<.0001
Material*Storage*time		Ref	5	3	56.2	89.48	<.0001

10. Method = TBA 10:21 Wednesday, March 28, 2001 22

The Mixed Procedure

Tests of Effect Slices

Effect	Material	Storage	time	Num DF	Den DF	F Value	Pr > F
Material*Storage*time		Ref	6	3	56.2	0.42	0.7425
Material*Storage*time		Ref	7	3	56.2	0.19	0.9029
Material*Storage*time	DK11		1	3	56.2	149.62	<.0001
Material*Storage*time	DK11		2	3	56.2	13.46	<.0001
Material*Storage*time	DK11		3	3	56.2	20.45	<.0001
Material*Storage*time	DK11		4	3	56.2	66.58	<.0001
Material*Storage*time	DK11		5	3	56.2	146.72	<.0001
Material*Storage*time	DK11		6	3	56.2	15.04	<.0001
Material*Storage*time	DK11		7	3	56.2	24.88	<.0001
Material*Storage*time	HB1		1	3	56.2	120.05	<.0001

Material*Storage*time	HB1		2	3	56.2	57.33	<.0001
Material*Storage*time	HB1		3	3	56.2	2.51	0.0682
Material*Storage*time	HB1		4	3	56.2	50.76	<.0001
Material*Storage*time	HB1		5	3	56.2	73.26	<.0001
Material*Storage*time	HB1		6	3	56.2	10.4	<.0001
Material*Storage*time	HB1		7	3	56.2	19.13	<.0001
Material*Storage*time	PrntPk		1	3	56.2	61.58	<.0001
Material*Storage*time	PrntPk		2	3	56.2	44.86	<.0001
Material*Storage*time	PrntPk		3	3	56.2	20.82	<.0001
Material*Storage*time	PrntPk		4	3	56.2	34.98	<.0001
Material*Storage*time	PrntPk		5	3	56.2	11.79	<.0001
Material*Storage*time	PrntPk		6	3	56.2	5.1	0.0034
Material*Storage*time	PrntPk		7	3	56.2	1.36	0.2632
Material*Storage*time	Saran		1	3	56.2	264.34	<.0001
Material*Storage*time	Saran		2	3	56.2	40.91	<.0001
Material*Storage*time	Saran		3	3	56.2	35.79	<.0001
Material*Storage*time	Saran		4	3	56.2	61	<.0001
Material*Storage*time	Saran		5	3	56.2	52.12	<.0001
Material*Storage*time	Saran		6	3	56.2	23.77	<.0001
Material*Storage*time	Saran		7	3	56.2	10.64	<.0001

Time = 1

The Mixed Procedure

Model Information

Data Set	WORK.NEW
Dependent Variable	petctrl2
Covariance Structure	Diagonal
Estimation Method	REML
Residual Variance Method	Profile
Fixed Effects SE Method	Model-Based
Degrees of Freedom Method	Residual

Class Level Information

Class	Levels	Values
Material	4	DK11 HB1 PrntPk Saran
Storage	4	HC O RT Ref

Dimensions

Covariance Parameters	1
Columns in X	25
Columns in Z	0
Subjects	1
Max Obs Per Subject	32
Observations Used	32
Observations Not Used	0
Total Observations	32

Covariance Parameter Estimates

Cov Parm	Estimate	Standard Error	Z Value	Pr Z
Residual	0.02536	0.008965	2.83	0.0023

Fit Statistics

-2 Res Log Likelihood	-2.3
AIC (smaller is better)	-0.3
AICC (smaller is better)	0
BIC (smaller is better)	0.5

Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Material	3	16	4.43	0.0189
Storage	3	16	4.97	0.0127
Material*Storage	9	16	5.47	0.0016

Time = 1

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	Estimate	Standard Error	DF	t Value	y
Material*Storage	DK11	HC	5.5791	0.1126	16	49.55	<.0001
Material*Storage	DK11	O	5.1274	0.1126	16	45.54	<.0001
Material*Storage	DK11	RT	5.6091	0.1126	16	49.82	<.0001
Material*Storage	DK11	Ref	5.7302	0.1126	16	50.89	<.0001
Material*Storage	HB1	HC	5.8884	0.1126	16	52.3	<.0001
Material*Storage	HB1	O	5.4788	0.1126	16	48.66	<.0001
Material*Storage	HB1	RT	5.6483	0.1126	16	50.16	<.0001
Material*Storage	HB1	Ref	5.7187	0.1126	16	50.79	<.0001
Material*Storage	PrntPk	HC	5.6932	0.1126	16	50.56	<.0001
Material*Storage	PrntPk	O	5.6585	0.1126	16	50.25	<.0001
Material*Storage	PrntPk	RT	5.2608	0.1126	16	46.72	<.0001
Material*Storage	PrntPk	Ref	5.0819	0.1126	16	45.13	<.0001
Material*Storage	Saran	HC	5.7893	0.1126	16	51.42	<.0001
Material*Storage	Saran	O	5.8804	0.1126	16	52.23	<.0001
Material*Storage	Saran	RT	5.2324	0.1126	16	46.47	<.0001
Material*Storage	Saran	Ref	5.6467	0.1126	16	50.15	<.0001

Tests of Effect Slices

Effect	Material	Storage	Num DF	Den DF	F Value	Pr > F
Material*Storage		HC	3	16	1.38	0.2846
Material*Storage		O	3	16	7.99	0.0018
Material*Storage		RT	3	16	3.87	0.0295
Material*Storage		Ref	3	16	7.61	0.0022
Material*Storage	DK11		3	16	5.51	0.0086
Material*Storage	HB1		3	16	2.27	0.1195
Material*Storage	PrntPk		3	16	7.13	0.003
Material*Storage	Saran		3	16	6.47	0.0045

Time = 1

Material	Storage	Material	Storage	Estimate	StdErr	DF	tValue	Probt
DK11	HC	DK11	O	0.4518	0.1592	16	2.84	0.0119
DK11	HC	DK11	RT	-0.02991	0.1592	16	-0.19	0.8534
DK11	HC	DK11	Ref	-0.1511	0.1592	16	-0.95	0.3568
DK11	HC	HB1	HC	-0.3093	0.1592	16	-1.94	0.0699
DK11	HC	HB1	O	0.1003	0.1592	16	0.63	0.5376
DK11	HC	HB1	RT	-0.06919	0.1592	16	-0.43	0.6697
DK11	HC	HB1	Ref	-0.1395	0.1592	16	-0.88	0.3938
DK11	HC	PrntPk	HC	-0.114	0.1592	16	-0.72	0.4842
DK11	HC	PrntPk	O	-0.07935	0.1592	16	-0.5	0.625
DK11	HC	PrntPk	RT	0.3183	0.1592	16	2	0.0629
DK11	HC	PrntPk	Ref	0.4973	0.1592	16	3.12	0.0066
DK11	HC	Saran	HC	-0.2102	0.1592	16	-1.32	0.2054
DK11	HC	Saran	O	-0.3013	0.1592	16	-1.89	0.0767
DK11	HC	Saran	RT	0.3467	0.1592	16	2.18	0.0448
DK11	HC	Saran	Ref	-0.06755	0.1592	16	-0.42	0.6771
DK11	O	DK11	RT	-0.4817	0.1592	16	-3.02	0.008
DK11	O	DK11	Ref	-0.6029	0.1592	16	-3.79	0.0016
DK11	O	HB1	HC	-0.7611	0.1592	16	-4.78	0.0002
DK11	O	HB1	O	-0.3515	0.1592	16	-2.21	0.0423
DK11	O	HB1	RT	-0.521	0.1592	16	-3.27	0.0048
DK11	O	HB1	Ref	-0.5913	0.1592	16	-3.71	0.0019
DK11	O	PrntPk	HC	-0.5658	0.1592	16	-3.55	0.0026
DK11	O	PrntPk	O	-0.5311	0.1592	16	-3.34	0.0042
DK11	O	PrntPk	RT	-0.1334	0.1592	16	-0.84	0.4144
DK11	O	PrntPk	Ref	0.04549	0.1592	16	0.29	0.7788
DK11	O	Saran	HC	-0.662	0.1592	16	-4.16	0.0007
DK11	O	Saran	O	-0.753	0.1592	16	-4.73	0.0002
DK11	O	Saran	RT	-0.105	0.1592	16	-0.66	0.5189
DK11	O	Saran	Ref	-0.5193	0.1592	16	-3.26	0.0049
DK11	RT	DK11	Ref	-0.1212	0.1592	16	-0.76	0.4577
DK11	RT	HB1	HC	-0.2794	0.1592	16	-1.75	0.0985
DK11	RT	HB1	O	0.1302	0.1592	16	0.82	0.4255
DK11	RT	HB1	RT	-0.03928	0.1592	16	-0.25	0.8083
DK11	RT	HB1	Ref	-0.1096	0.1592	16	-0.69	0.501
DK11	RT	PrntPk	HC	-0.08413	0.1592	16	-0.53	0.6045
DK11	RT	PrntPk	O	-0.04944	0.1592	16	-0.31	0.7602
DK11	RT	PrntPk	RT	0.3482	0.1592	16	2.19	0.0439
DK11	RT	PrntPk	Ref	0.5272	0.1592	16	3.31	0.0044
DK11	RT	Saran	HC	-0.1803	0.1592	16	-1.13	0.2742
DK11	RT	Saran	O	-0.2714	0.1592	16	-1.7	0.1077

DK11	RT	Saran	RT	0.3766	0.1592	16	2.37	0.031
DK11	RT	Saran	Ref	-0.03764	0.1592	16	-0.24	0.8161
DK11	Ref	HB1	HC	-0.1582	0.1592	16	-0.99	0.3353
DK11	Ref	HB1	O	0.2514	0.1592	16	1.58	0.1339
DK11	Ref	HB1	RT	0.08191	0.1592	16	0.51	0.614
DK11	Ref	HB1	Ref	0.01156	0.1592	16	0.07	0.943
DK11	Ref	PrntPk	HC	0.03706	0.1592	16	0.23	0.8189
DK11	Ref	PrntPk	O	0.07175	0.1592	16	0.45	0.6584
DK11	Ref	PrntPk	RT	0.4694	0.1592	16	2.95	0.0094
DK11	Ref	PrntPk	Ref	0.6484	0.1592	16	4.07	0.0009
DK11	Ref	Saran	HC	-0.0591	0.1592	16	-0.37	0.7154
DK11	Ref	Saran	O	-0.1502	0.1592	16	-0.94	0.3597
DK11	Ref	Saran	RT	0.4978	0.1592	16	3.13	0.0065
DK11	Ref	Saran	Ref	0.08355	0.1592	16	0.52	0.607
HB1	HC	HB1	O	0.4096	0.1592	16	2.57	0.0205
HB1	HC	HB1	RT	0.2401	0.1592	16	1.51	0.1511
HB1	HC	HB1	Ref	0.1698	0.1592	16	1.07	0.3022
HB1	HC	PrntPk	HC	0.1953	0.1592	16	1.23	0.2378
HB1	HC	PrntPk	O	0.2299	0.1592	16	1.44	0.168
HB1	HC	PrntPk	RT	0.6276	0.1592	16	3.94	0.0012
HB1	HC	PrntPk	Ref	0.8066	0.1592	16	5.07	0.0001
HB1	HC	Saran	HC	0.0991	0.1592	16	0.62	0.5425

Time = 1

Material	Storage	Material	Storage	Estimate	StdErr	DF	tValue	Probt
HB1	HC	Saran	O	0.008034	0.1592	16	0.05	0.9604
HB1	HC	Saran	RT	0.656	0.1592	16	4.12	0.0008
HB1	HC	Saran	Ref	0.2417	0.1592	16	1.52	0.1485
HB1	O	HB1	RT	-0.1695	0.1592	16	-1.06	0.3029
HB1	O	HB1	Ref	-0.2399	0.1592	16	-1.51	0.1515
HB1	O	PrntPk	HC	-0.2143	0.1592	16	-1.35	0.197
HB1	O	PrntPk	O	-0.1797	0.1592	16	-1.13	0.2758
HB1	O	PrntPk	RT	0.218	0.1592	16	1.37	0.1899
HB1	O	PrntPk	Ref	0.397	0.1592	16	2.49	0.024
HB1	O	Saran	HC	-0.3105	0.1592	16	-1.95	0.0689
HB1	O	Saran	O	-0.4016	0.1592	16	-2.52	0.0227
HB1	O	Saran	RT	0.2464	0.1592	16	1.55	0.1413
HB1	O	Saran	Ref	-0.1679	0.1592	16	-1.05	0.3075
HB1	RT	HB1	Ref	-0.07035	0.1592	16	-0.44	0.6645
HB1	RT	PrntPk	HC	-0.04485	0.1592	16	-0.28	0.7818
HB1	RT	PrntPk	O	-0.01017	0.1592	16	-0.06	0.9499

HB1	RT	PrntPk	RT	0.3875	0.1592	16	2.43	0.027
HB1	RT	PrntPk	Ref	0.5665	0.1592	16	3.56	0.0026
HB1	RT	Saran	HC	-0.141	0.1592	16	-0.89	0.389
HB1	RT	Saran	O	-0.2321	0.1592	16	-1.46	0.1643
HB1	RT	Saran	RT	0.4159	0.1592	16	2.61	0.0189
HB1	RT	Saran	Ref	0.001638	0.1592	16	0.01	0.9919
HB1	Ref	PrntPk	HC	0.02551	0.1592	16	0.16	0.8747
HB1	Ref	PrntPk	O	0.06019	0.1592	16	0.38	0.7104
HB1	Ref	PrntPk	RT	0.4579	0.1592	16	2.88	0.011
HB1	Ref	PrntPk	Ref	0.6368	0.1592	16	4	0.001
HB1	Ref	Saran	HC	-0.07066	0.1592	16	-0.44	0.6632
HB1	Ref	Saran	O	-0.1617	0.1592	16	-1.02	0.3249
HB1	Ref	Saran	RT	0.4863	0.1592	16	3.05	0.0076
HB1	Ref	Saran	Ref	0.07199	0.1592	16	0.45	0.6573
PrntPk	HC	PrntPk	O	0.03468	0.1592	16	0.22	0.8303
PrntPk	HC	PrntPk	RT	0.4324	0.1592	16	2.72	0.0153
PrntPk	HC	PrntPk	Ref	0.6113	0.1592	16	3.84	0.0014
PrntPk	HC	Saran	HC	-0.09616	0.1592	16	-0.6	0.5544
PrntPk	HC	Saran	O	-0.1872	0.1592	16	-1.18	0.2569
PrntPk	HC	Saran	RT	0.4608	0.1592	16	2.89	0.0106
PrntPk	HC	Saran	Ref	0.04649	0.1592	16	0.29	0.7741
PrntPk	O	PrntPk	RT	0.3977	0.1592	16	2.5	0.0238
PrntPk	O	PrntPk	Ref	0.5766	0.1592	16	3.62	0.0023
PrntPk	O	Saran	HC	-0.1308	0.1592	16	-0.82	0.4233
PrntPk	O	Saran	O	-0.2219	0.1592	16	-1.39	0.1825
PrntPk	O	Saran	RT	0.4261	0.1592	16	2.68	0.0166
PrntPk	O	Saran	Ref	0.0118	0.1592	16	0.07	0.9418
PrntPk	RT	PrntPk	Ref	0.1789	0.1592	16	1.12	0.2777
PrntPk	RT	Saran	HC	-0.5285	0.1592	16	-3.32	0.0043
PrntPk	RT	Saran	O	-0.6196	0.1592	16	-3.89	0.0013
PrntPk	RT	Saran	RT	0.0284	0.1592	16	0.18	0.8607
PrntPk	RT	Saran	Ref	-0.3859	0.1592	16	-2.42	0.0276
PrntPk	Ref	Saran	HC	-0.7075	0.1592	16	-4.44	0.0004
PrntPk	Ref	Saran	O	-0.7985	0.1592	16	-5.01	0.0001
PrntPk	Ref	Saran	RT	-0.1505	0.1592	16	-0.95	0.3585
PrntPk	Ref	Saran	Ref	-0.5648	0.1592	16	-3.55	0.0027
Saran	HC	Saran	O	-0.09107	0.1592	16	-0.57	0.5753
Saran	HC	Saran	RT	0.5569	0.1592	16	3.5	0.003
Saran	HC	Saran	Ref	0.1426	0.1592	16	0.9	0.3836
Saran	O	Saran	RT	0.648	0.1592	16	4.07	0.0009
Saran	O	Saran	Ref	0.2337	0.1592	16	1.47	0.1616
Saran	RT	Saran	Ref	-0.4143	0.1592	16	-2.6	0.0193

Time = 1

The Mixed Procedure

Model Information

Data Set	WORK.NEW
Dependent Variable	pctctrl2
Covariance Structure	Diagonal
Estimation Method	REML
Residual Variance Method	Profile
Fixed Effects SE Method	Model-Based
Degrees of Freedom Method	Residual

Class Level Information

Class	Levels	Values
Material	4	DK11 HB1 PrntPk Saran
Storage	4	HC O RT Ref

Dimensions

Covariance Parameters	1
Columns in X	25
Columns in Z	0
Subjects	1
Max Obs Per Subject	32
Observations Used	32
Observations Not Used	0
Total Observations	32

Covariance Parameter Estimates

Cov Parm	Estimate	Standard Error	Z Value	Pr Z
Residual	0.006409	0.002266	2.83	0.0023

Fit Statistics

-2 Res Log Likelihood	-24.3
AIC (smaller is better)	-22.3
AICC (smaller is better)	-22
BIC (smaller is better)	-21.5

Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Material	3	16	134.48	<.0001
Storage	3	16	722.29	<.0001
Material*Storage	9	16	55.98	<.0001

Time = 1

The Mixed Procedure

Least Squares Means

Effect	Material	Storage	Estimate	Standard Error	DF	t Value	Pr > |t|
Material*Storage	DK11	HC	6.8992	0.05661	16	121.87	<.0001
Material*Storage	DK11	O	6.0382	0.05661	16	106.66	<.0001
Material*Storage	DK11	RT	7.132	0.05661	16	125.99	<.0001
Material*Storage	DK11	Ref	5.2822	0.05661	16	93.31	<.0001
Material*Storage	HB1	HC	6.1959	0.05661	16	109.45	<.0001
Material*Storage	HB1	O	6.3574	0.05661	16	112.3	<.0001
Material*Storage	HB1	RT	6.8473	0.05661	16	120.96	<.0001
Material*Storage	HB1	Ref	5.055	0.05661	16	89.3	<.0001
Material*Storage	PrntPk	HC	6.2561	0.05661	16	110.51	<.0001
Material*Storage	PrntPk	O	5.8336	0.05661	16	103.05	<.0001
Material*Storage	PrntPk	RT	5.453	0.05661	16	96.33	<.0001
Material*Storage	PrntPk	Ref	4.9819	0.05661	16	88	<.0001
Material*Storage	Saran	HC	7.1822	0.05661	16	126.87	<.0001

Material*Storage	Saran	O	6.4734	0.05661	16	114.35	<.0001
Material*Storage	Saran	RT	6.9218	0.05661	16	122.27	<.0001
Material*Storage	Saran	Ref	4.6861	0.05661	16	82.78	<.0001

Tests of Effect Slices

Effect	Material	Storage	Num DF	Den DF	F Value	Pr > F
Material*Storage		HC	3	16	73.39	<.0001
Material*Storage		O	3	16	26.79	<.0001
Material*Storage		RT	3	16	183.36	<.0001
Material*Storage		Ref	3	16	18.88	<.0001
Material*Storage	DK11		3	16	223.64	<.0001
Material*Storage	HB1		3	16	179.44	<.0001
Material*Storage	PrntPk		3	16	92.04	<.0001
Material*Storage	Saran		3	16	395.11	<.0001

Time = 1

Material	Storage	Material	Storage	Estimate	StdErr	DF	tValue	Probt
DK11	HC	DK11	O	0.861	0.08006	16	10.76	<.0001
DK11	HC	DK11	RT	-0.2328	0.08006	16	-2.91	0.0103
DK11	HC	DK11	Ref	1.617	0.08006	16	20.2	<.0001
DK11	HC	HB1	HC	0.7033	0.08006	16	8.78	<.0001
DK11	HC	HB1	O	0.5419	0.08006	16	6.77	<.0001
DK11	HC	HB1	RT	0.05193	0.08006	16	0.65	0.5258
DK11	HC	HB1	Ref	1.8442	0.08006	16	23.04	<.0001
DK11	HC	PrntPk	HC	0.6431	0.08006	16	8.03	<.0001
DK11	HC	PrntPk	O	1.0657	0.08006	16	13.31	<.0001
DK11	HC	PrntPk	RT	1.4462	0.08006	16	18.06	<.0001
DK11	HC	PrntPk	Ref	1.9174	0.08006	16	23.95	<.0001
DK11	HC	Saran	HC	-0.283	0.08006	16	-3.53	0.0028
DK11	HC	Saran	O	0.4259	0.08006	16	5.32	<.0001
DK11	HC	Saran	RT	-0.02252	0.08006	16	-0.28	0.7821
DK11	HC	Saran	Ref	2.2131	0.08006	16	27.64	<.0001
DK11	O	DK11	RT	-1.0938	0.08006	16	-13.66	<.0001
DK11	O	DK11	Ref	0.756	0.08006	16	9.44	<.0001
DK11	O	HB1	HC	-0.1577	0.08006	16	-1.97	0.0664
DK11	O	HB1	O	-0.3192	0.08006	16	-3.99	0.0011
DK11	O	HB1	RT	-0.8091	0.08006	16	-10.11	<.0001
DK11	O	HB1	Ref	0.9832	0.08006	16	12.28	<.0001
DK11	O	PrntPk	HC	-0.2179	0.08006	16	-2.72	0.0151
DK11	O	PrntPk	O	0.2046	0.08006	16	2.56	0.0211

DK11	O	PrntPk	RT	0.5852	0.08006	16	7.31	<.0001
DK11	O	PrntPk	Ref	1.0563	0.08006	16	13.19	<.0001
DK11	O	Saran	HC	-1.144	0.08006	16	-14.29	<.0001
DK11	O	Saran	O	-0.4352	0.08006	16	-5.44	<.0001
DK11	O	Saran	RT	-0.8835	0.08006	16	-11.04	<.0001
DK11	O	Saran	Ref	1.3521	0.08006	16	16.89	<.0001
DK11	RT	DK11	Ref	1.8498	0.08006	16	23.11	<.0001
DK11	RT	HB1	HC	0.9361	0.08006	16	11.69	<.0001
DK11	RT	HB1	O	0.7747	0.08006	16	9.68	<.0001
DK11	RT	HB1	RT	0.2847	0.08006	16	3.56	0.0026
DK11	RT	HB1	Ref	2.077	0.08006	16	25.94	<.0001
DK11	RT	PrntPk	HC	0.8759	0.08006	16	10.94	<.0001
DK11	RT	PrntPk	O	1.2985	0.08006	16	16.22	<.0001
DK11	RT	PrntPk	RT	1.679	0.08006	16	20.97	<.0001
DK11	RT	PrntPk	Ref	2.1502	0.08006	16	26.86	<.0001
DK11	RT	Saran	HC	-0.05019	0.08006	16	-0.63	0.5395
DK11	RT	Saran	O	0.6587	0.08006	16	8.23	<.0001
DK11	RT	Saran	RT	0.2103	0.08006	16	2.63	0.0183
DK11	RT	Saran	Ref	2.4459	0.08006	16	30.55	<.0001
DK11	Ref	HB1	HC	-0.9137	0.08006	16	-11.41	<.0001
DK11	Ref	HB1	O	-1.0752	0.08006	16	-13.43	<.0001
DK11	Ref	HB1	RT	-1.5651	0.08006	16	-19.55	<.0001
DK11	Ref	HB1	Ref	0.2272	0.08006	16	2.84	0.0119
DK11	Ref	PrntPk	HC	-0.974	0.08006	16	-12.17	<.0001
DK11	Ref	PrntPk	O	-0.5514	0.08006	16	-6.89	<.0001
DK11	Ref	PrntPk	RT	-0.1708	0.08006	16	-2.13	0.0487
DK11	Ref	PrntPk	Ref	0.3003	0.08006	16	3.75	0.0017
DK11	Ref	Saran	HC	-1.9	0.08006	16	-23.73	<.0001
DK11	Ref	Saran	O	-1.1912	0.08006	16	-14.88	<.0001
DK11	Ref	Saran	RT	-1.6396	0.08006	16	-20.48	<.0001
DK11	Ref	Saran	Ref	0.5961	0.08006	16	7.45	<.0001
HB1	HC	HB1	O	-0.1614	0.08006	16	-2.02	0.0609
HB1	HC	HB1	RT	-0.6514	0.08006	16	-8.14	<.0001
HB1	HC	HB1	Ref	1.1409	0.08006	16	14.25	<.0001
HB1	HC	PrntPk	HC	-0.06021	0.08006	16	-0.75	0.4629
HB1	HC	PrntPk	O	0.3624	0.08006	16	4.53	0.0003
HB1	HC	PrntPk	RT	0.7429	0.08006	16	9.28	<.0001
HB1	HC	PrntPk	Ref	1.2141	0.08006	16	15.16	<.0001
HB1	HC	Saran	HC	-0.9863	0.08006	16	-12.32	<.0001

Time = 1

Material	Storage	Material	Storage	Estimate	StdErr	DF	tValue	Probt
HB1	HC	Saran	O	-0.2774	0.08006	16	-3.47	0.0032
HB1	HC	Saran	RT	-0.7258	0.08006	16	-9.07	<.0001
HB1	HC	Saran	Ref	1.5098	0.08006	16	18.86	<.0001
HB1	O	HB1	RT	-0.4899	0.08006	16	-6.12	<.0001
HB1	O	HB1	Ref	1.3023	0.08006	16	16.27	<.0001
HB1	O	PrntPk	HC	0.1012	0.08006	16	1.26	0.2242
HB1	O	PrntPk	O	0.5238	0.08006	16	6.54	<.0001
HB1	O	PrntPk	RT	0.9044	0.08006	16	11.3	<.0001
HB1	O	PrntPk	Ref	1.3755	0.08006	16	17.18	<.0001
HB1	O	Saran	HC	-0.8249	0.08006	16	-10.3	<.0001
HB1	O	Saran	O	-0.116	0.08006	16	-1.45	0.1666
HB1	O	Saran	RT	-0.5644	0.08006	16	-7.05	<.0001
HB1	O	Saran	Ref	1.6713	0.08006	16	20.88	<.0001
HB1	RT	HB1	Ref	1.7923	0.08006	16	22.39	<.0001
HB1	RT	PrntPk	HC	0.5912	0.08006	16	7.38	<.0001
HB1	RT	PrntPk	O	1.0137	0.08006	16	12.66	<.0001
HB1	RT	PrntPk	RT	1.3943	0.08006	16	17.42	<.0001
HB1	RT	PrntPk	Ref	1.8654	0.08006	16	23.3	<.0001
HB1	RT	Saran	HC	-0.3349	0.08006	16	-4.18	0.0007
HB1	RT	Saran	O	0.3739	0.08006	16	4.67	0.0003
HB1	RT	Saran	RT	-0.07444	0.08006	16	-0.93	0.3663
HB1	RT	Saran	Ref	2.1612	0.08006	16	27	<.0001
HB1	Ref	PrntPk	HC	-1.2011	0.08006	16	-15	<.0001
HB1	Ref	PrntPk	O	-0.7785	0.08006	16	-9.72	<.0001
HB1	Ref	PrntPk	RT	-0.398	0.08006	16	-4.97	0.0001
HB1	Ref	PrntPk	Ref	0.07313	0.08006	16	0.91	0.3745
HB1	Ref	Saran	HC	-2.1272	0.08006	16	-26.57	<.0001
HB1	Ref	Saran	O	-1.4184	0.08006	16	-17.72	<.0001
HB1	Ref	Saran	RT	-1.8667	0.08006	16	-23.32	<.0001
HB1	Ref	Saran	Ref	0.3689	0.08006	16	4.61	0.0003
PrntPk	HC	PrntPk	O	0.4226	0.08006	16	5.28	<.0001
PrntPk	HC	PrntPk	RT	0.8032	0.08006	16	10.03	<.0001
PrntPk	HC	PrntPk	Ref	1.2743	0.08006	16	15.92	<.0001
PrntPk	HC	Saran	HC	-0.9261	0.08006	16	-11.57	<.0001
PrntPk	HC	Saran	O	-0.2172	0.08006	16	-2.71	0.0153
PrntPk	HC	Saran	RT	-0.6656	0.08006	16	-8.31	<.0001
PrntPk	HC	Saran	Ref	1.57	0.08006	16	19.61	<.0001
PrntPk	O	PrntPk	RT	0.3806	0.08006	16	4.75	0.0002
PrntPk	O	PrntPk	Ref	0.8517	0.08006	16	10.64	<.0001

PrntPk	O	Saran	HC	-1.3487	0.08006	16	-16.85	<.0001
PrntPk	O	Saran	O	-0.6398	0.08006	16	-7.99	<.0001
PrntPk	O	Saran	RT	-1.0882	0.08006	16	-13.59	<.0001
PrntPk	O	Saran	Ref	1.1474	0.08006	16	14.33	<.0001
PrntPk	RT	PrntPk	Ref	0.4711	0.08006	16	5.88	<.0001
PrntPk	RT	Saran	HC	-1.7292	0.08006	16	-21.6	<.0001
PrntPk	RT	Saran	O	-1.0204	0.08006	16	-12.75	<.0001
PrntPk	RT	Saran	RT	-1.4688	0.08006	16	-18.35	<.0001
PrntPk	RT	Saran	Ref	0.7669	0.08006	16	9.58	<.0001
PrntPk	Ref	Saran	HC	-2.2003	0.08006	16	-27.48	<.0001
PrntPk	Ref	Saran	O	-1.4915	0.08006	16	-18.63	<.0001
PrntPk	Ref	Saran	RT	-1.9399	0.08006	16	-24.23	<.0001
PrntPk	Ref	Saran	Ref	0.2958	0.08006	16	3.69	0.002
Saran	HC	Saran	O	0.7089	0.08006	16	8.85	<.0001
Saran	HC	Saran	RT	0.2605	0.08006	16	3.25	0.005
Saran	HC	Saran	Ref	2.4961	0.08006	16	31.18	<.0001
Saran	O	Saran	RT	-0.4484	0.08006	16	-5.6	<.0001
Saran	O	Saran	Ref	1.7873	0.08006	16	22.32	<.0001
Saran	RT	Saran	Ref	2.2356	0.08006	16	27.93	<.0001

VITA

Mimin Adhikary

Candidate for the Degree of

Master of Science

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